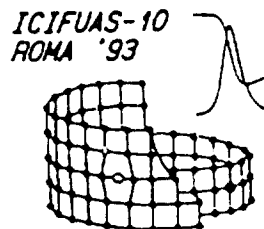


10th International Conference on

ADA 276 849

INTERNAL FRICTION AND ULTRASONIC ATTENUATION IN SOLIDS -  
ICIFUAS-10

Roma (Italy), September 6-9, 1993



SPONSORS

Università di Roma "La Sapienza"  
Dipartimenti di Fisica & Energetica  
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American Physical Society  
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Comune di Roma - Assessorato alla Cultura  
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Background

The Conference is the 10th of a quadrennial series which began in Providence (USA) in 1956 and continued in Ithaca (USA, 1961), Manchester (England, 1965), Providence (USA, 1969), Aachen (Germany, 1973), Tokyo (Japan, 1977), Lausanne (Switzerland, 1981), Urbana (USA, 1985), and Beijing (China, 1989).

Purpose

To provide an International Forum for researchers in the field of elastic energy dissipation in solids by ultrasonic attenuation, internal friction, after effect, and vibration damping.

**Best  
Available  
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### International Scientific Committee

R.D. Adams	Bristol (England)
D.P. Almond	Bath (England)
W. Benoit	Lausanne (Switzerland)
G. Cannelli	Roma (Italy)
R. Cantelli	Roma (Italy)
T.G. Chen	Shanghai (China)
R. De Batist	Antwerpen (Belgium)
J. de Fouquet	Poitiers (France)
C. Elbaum	Providence (USA)
G. Fantozzi	Villeurbanne (France)
A.V. Granato	Urbana (USA)
S. Hunklinger	Heidelberg (Germany)
S. Kiss	Debrecen (Hungary)
M. Koiwa	Kyoto (Japan)
M. Levy	Milwaukee (USA)
L.B. Magalas	Cracow (Poland)
V. Müller	Berlin (Germany)
S.P. Nikanorov	S. Petersburg (Russia)
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I.G. Ritchie	Pinawa (Canada)
D. Sette	Roma (Italy)
Y. Wang	Nanjing (China)
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Conference Chairman: R. Cantelli

### International Honorary Committee

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A. Seeger	Stuttgart (Germany)
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L. Verdini	Perugia

### Topics

1. Point defects.
2. Dislocations and their interaction with lattice imperfections.
3. Phase transformations: a) displacive transitions; b) order-disorder transitions and precipitation.
4. Hydrogen in metals and semiconductors.
5. Oxide superconductors and fullerenes.
6. Amorphous materials and superionic conductors.
7. Ferroelectric and ceramic materials.
8. Composites and polymers.
9. Thin films.
10. Electron and phonon relaxation.
11. Grain boundaries, domain walls, and interfaces.
12. General aspects of anelasticity and mechanical properties.
13. Magneto-elastic interactions.
14. Experimental techniques, non-destructive testing, industrial applications.

# PROGRAMME

**SUNDAY 5th (15.00 - 20.00) and MONDAY 6th (from 8.00)**

## REGISTRATION

Secretariat Room

**MONDAY September 6th 1993**

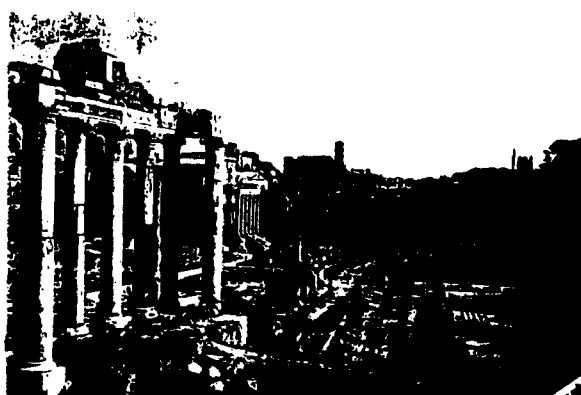
OPENING Aula Magna		
8.40	<i>G. Tecce (Chancellor of the University of Rome "La Sapienza")</i> <i>G. Salvini (President of "Accademia Nazionale dei Lincei")</i> <i>R. Cantelli (Chairman of "ICIFUAS 10")</i>	
9.10	<i>A. Seeger</i> 40 years of dislocation interpretation of the Bordoni relaxation - solved and unsolved problems.	
	1 - POINT DEFECTS Chairman: A.S. Nowick Lecture Room 1	8 - COMPOSITES AND POLYMERS Chairman: G. Fantozzi Lecture Room 2
10.00	<i>P. Esquinazi*</i> O1-1 Internal friction and sound velocity of polycrystals at very low temperature	<i>R. Schaller</i> O8-1 Influence of changing microstructure on high temperature relaxation peaks, an example WC-11wt%Co
10.25	Coffee break	
10.45	<i>G. Haneczock</i> O1-2 Mechanical loss studies of oxygen-oxygen interaction in tantalum	<i>C.M. Langton</i> O8-2 Identification and characterization of reclaimed and recycled polymers by ultrasound attenuation
11.10	<i>O. Florencio</i> O1-3 Anelastic behaviour in Nb-Ti alloys containing interstitial elements	<i>D.P. Almond*</i> O8-3 Dynamic mechanical analysis of glass transition peaks in polymers and composites
11.35	<i>H. Tanimoto</i> O1-4 Migration mechanism of self-interstitial atoms in Mo after low temperature irradiation	<i>C. Wert*</i> O8-4 JET: historical, physical and material aspects
12.00	<i>A. Biscarini</i> O1-5 Interstitial site occupancies and hydrogen transition probabilities in FCC binary alloys: statistical model	<i>A. Vincent</i> O8-5 Transient internal damping in aluminium based metal matrix composites
12.25	Lunch	

\* invited lectures

	<b>4 - HYDROGEN IN METALS AND SEMICONDUCTORS</b>	<b>9-THIN FILMS</b>	
	<b>Chairman: H. Wipf</b>	<b>Chairman: S. Okuda</b>	
	<b>Lecture Room 1</b>	<b>Lecture Room 2</b>	
14.00	<i>H.R. Sinning*</i> <b>O4-1</b> Mechanical spectroscopy with hydrogen in intermetallic phases	<i>H.G. Bohn*</i> <b>O9-1</b> Grain boundary sliding in thin substrate-bonded Al-films	
14.25	<i>M. Ege</i> <b>O4-2</b> Reorientation of biatomic H-Y complexes in diluted Pd/Y alloys	<i>J.F. Scott</i> <b>O9-2</b> Loss mechanisms in fine-grained ferroelectric ceramic thin films for ULSI memories (DRAMs)	
14.50	<i>F. Cordero</i> <b>O4-3</b> Four-site tunnelling of H trapped by substitutional Zr in Nb	<i>O. Yoshinari</i> <b>O9-3</b> Internal friction and elasticity of V and Cr films on Si-substrates	
15.15	<i>Coffee break</i>		
	<b>POSTER SESSION I</b>		
15.30	<b>Presentation</b> topics: 1, 4 and 12 Chairman: M. Levy <b>Lecture room 1</b>	<b>Presentation</b> topics: 6, 8 and 9 Chairman: G. Weiss <b>Lecture room 2</b>	<b>Presentation</b> topics: 2 Chairman: M. Weller <b>Lecture room 3</b>
17.00	<b>VISIT TO POSTERS</b>		
18.30	(Topics 1, 2, 4, 6, 8, 9 and 12)		

19.15

### *Conference Reception in Villa Caffarelli*



Villa Caffarelli is a 16th century building which, together with the "Palazzo Senatorio", "Palazzo dei Conservatori" and "Palazzo del Museo Capitolino", constitutes the City Hall of Rome located on the Capitolium Hill.

From the Caffarelli Terrace it is possible to enjoy a view of Rome and especially the Roman Forum.

## TUESDAY September 7th 1993

8.50 9.00.	<b>Announcements</b>	
	<b>Secretariat Room</b>	
	<b>5 - OXIDE SUPERCONDUCTORS AND FULLERENES</b>  Chairman: Y. Wang  <b>Lecture Room 1</b>	<b>2 - DISLOCATIONS AND THEIR INTERACTIONS WITH LATTICE IMPERFECTIONS</b>  Chairman: A. Seeger  <b>Lecture Room 2</b>
9.00	<i>R. Cantelli</i> 05-1  Oxygen diffusion and phase transformations in $\text{RBa}_2\text{Cu}_3\text{O}_{6+x}$	<i>T. Kosugi*</i> 02-1  The problem of the Peierls potential in FCC metals
9.25	<i>P. Devos</i> 05-2  Low temperature torsion pendulum measurements on YBCO superconductors	<i>W. Ulfert*</i> 02-2  Bordoni and hydrogen Snoek-Köster relaxation in fcc metals
9.50	<i>K. Fossheim*</i> 05-3  Ultrasonic anomalies and phase transitions in the $\text{Y}_1\text{Ba}_2\text{Cu}_4\text{O}_8$ and $\text{Y}_{1-x}\text{Ca}_x\text{Ba}_2\text{Cu}_4\text{O}_8$ systems	<i>V.P. Kisel*</i> 02-3  The dislocation-crystal defects interactions at extremely low and high temperatures
10.15	<i>Coffee break</i>	
	<b>5 - OXIDE SUPERCONDUCTORS AND FULLERENES</b>  Chairman: K. Fossheim  <b>Lecture Room 1</b>	<b>2 - DISLOCATIONS AND THEIR INTERACTIONS WITH LATTICE IMPERFECTIONS</b>  Chairman: G. Schoeck  <b>Lecture Room 2</b>
10.45	<i>H. Wipf*</i> 05-4  Elastic after-effect study of the ferroelastic tetragonal-orthorhombic phase transition in $\text{YBa}_2\text{Cu}_3\text{O}_x$	<i>F. Marchesoni</i> 02-4  Self-organized criticality and dislocation damping
11.10	<i>W. Grill</i> 05-5  Piezoelectric effect and ultrasonic attenuation near $T_c$ in single crystalline $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ superconductors	<i>A. Rivière</i> 02-5  High temperature internal friction in aluminium studied by isothermal mechanical spectrometry
11.35	<i>C. Hucho</i> 05-6  Ultrasonic investigations of dielectric properties of $\text{YBa}_2\text{Cu}_3\text{O}_{7.8}$	<i>S.B. Kustov</i> 02-6  The amplitude-dependent internal friction and dislocation inelastic strain in zinc and aluminium single crystals
12.00	<i>M. Levy*</i> 05-7  Superconducting energy gap in melt textured $\text{YBa}_2\text{Cu}_3\text{O}_7$ from low field ultrasonic measurements	<i>H. Numakura</i> 02-7  Internal friction due to twinning dislocations in Cu-Ge solid solution alloys
12.25	<i>Lunch</i>	

13.45

**Departure for Ostia Antica**

Ostia lies at the mouth of the river Tiber. It was founded by Ancus Marcius, 4th King of Rome, around 600 B.C. at the beginning of Rome's commercial, political and maritime expansion. Originally Ostia was a Castrum or fortified military base housing a garrison to defend navigation on the Tiber.



During the Republican period the Romans, after having consolidated their first conquests, transformed Ostia into a military port and into a real city serving as a commercial centre for Rome. Thus Ostia became the emporium of Rome. Enormous warehouses (Horrea) were built for goods arriving from overseas. The accumulating of goods and wealth caused the city to grow rapidly and luxuriously. Magnificent palaces, rows of shops, temples, the theatre and the Forum were built. During Imperial times Ostia became more and more important until reaching fifty thousand people under Hadrian in 200 A.D. After this period the city slowly declined; by the Middle Ages it was completely depopulated.

Today the extensive ruins of Ostia, in a impressive park of umbrella pines and cypresses give a remarkable idea of the domestic and commercial Roman architecture, and the way of life of people that lived there. So during the visit you will be able to satisfy your curiosity or simply enjoy the countryside.

18.30

**Dinner in the University Restaurant**

20.00

**Historical Reviews****"Physics of Anelasticity - the Early Days"**

Chairman: *K. Lücke*

*C. Wert*: Remembrances of Clarence Zener.

*A.S. Nowick*: Early work on dislocations and point defects.

*T.S. Kê*: Development of the torsion pendulum and the research on grain boundary relaxation and cold-work internal friction peak.

*P.G. Bordoni*: Dislocation relaxation.

**Aula Magna**

22.00

**Concert in Aula Magna**

*M. Campanella* (pianist) , *D. Beronesi* (mezzosoprano)

Organized by Prof. C. Rocca  
Director General of the Ministry of Tourism and Performing Arts

# WEDNESDAY September 8th 1993

8.50 9.00.	<b>Announcements</b>	
	<b>Secretariat Room</b>	
	<b>2 - DISLOCATIONS AND THEIR INTERACTIONS WITH LATTICE IMPERFECTIONS</b>  Chairman: H. Schultz  <b>Lecture Room 1</b>	<b>10 - ELECTRON AND PHONON RELAXATION</b>  Chairman: A.V. Granato  <b>Lecture Room 2</b>
9.00	<i>D.N. Beshers</i> O2-8  Nonlinear damping and harmonic generation in iron	<i>V.D. Fil</i> O10-1  Excitation and properties of zero sound in metals
9.25	<i>I.S. Golovin</i> O2-9  Internal friction and defect of modulus in a-Fe based high alloyed (Cr, Mo) hidamets	<i>D. Maurer*</i> O10-2  Unusual elastic behaviour of normal state UPt <sub>3</sub>
9.50	<i>S.P. Nikanorov *</i> O2-10  Amplitude-dependent internal friction and plastic properties of crystals	<i>P.P. Pal-Val</i> O10-3  Low-temperature anomalies of elastic moduli in niobium of different purity in the normal and superconducting states
10.15	<i>Coffee break</i>	
	<b>7 - FERROELECTRICS AND CERAMIC MATERIALS</b>  Chairman: G. Sorge  <b>Lecture Room 1</b>	<b>6 - AMORPHOUS MATERIALS AND SUPERIONIC CONDUCTORS</b>  Chairman: W. Arnold  <b>Lecture Room 2</b>
10.45	<i>G. Fantozzi*</i> O7-1  Anelastic behavior of barium titanate based ceramic materials	<i>G. Weiss*</i> O6-1  Low energy excitations in amorphous materials - acoustic experiments and comparison with theoretical models
11.10	<i>K. Matsushita</i> O7-2  Internal friction in ceria ceramics doped with alkali earth metal oxides	<i>K.L. Ngai*</i> O6-2  Theoretical basis and general applicability of the coupling model to relaxations in systems involving correlated motions: amorphous polymers, glassy ionic conductors, precipitates and cold worked metals (Snoek-Koster relaxation)
11.35	<i>Y.N. Wang*</i> O7-3  Mechanical and dielectric loss related to ferroelectrics and relaxor ferroelectrics phase transitions	<i>H. Mizubayashi</i> O6-3  Effect of passing electric current on the elastic properties of amorphous alloys
12.00	<i>G.V. Eynatten</i> O7-4  Change of sign of the piezoelectricity in BaTiO <sub>3</sub> -PMMA composites with frequency and temperature	<i>Y. Iliki</i> O6-4  Anelasticity and viscosity of superionic conducting glasses
12.25	<i>Lunch</i>	

	<b>13 - MAGNETO-ELASTIC INTERACTIONS</b>  Chairman: D.P. Almond  <b>Lecture Room 1</b>	<b>3 - PHASE TRANSFORMATIONS</b>  Chairman: A. Rivière  <b>Lecture Room 2</b>	
14.00	<b>V.J. Alshits*</b> O13-1  Magnetoplastic effect in non-magnetic crystals and internal friction	<b>G. Sörge*</b> O3-1  Investigations of the elastic behaviour in the vicinity of ferroelectric phase transitions	
14.25	<b>H. Uchida</b> O13-2  Giant-magnetostrictive materials: thin film formation and application to magnetic surface acoustic wave devices	<b>I. Yoshida</b> O3-2  Internal friction of Fe-Mn-Si alloy	
14.50	<b>L.E. Svistov</b> O13-3  Effect of high-frequency phonons on the magnetic moment of FeBO <sub>3</sub>	<b>A.L. Korzhenevskii</b> O3-3  The local phase transitions on mobile dislocations in crystals	
15.15	<i>Coffee break</i>		
15.35	<b>G. Huicheng</b> O13-4  The ultrasonic behaviour of ferrimagnetic garnet YIG crystals under pressure	<b>H. Shen</b> O3-4  Studies on pre-bainitic transformation in Cu-Zn-Al-Mn alloy	
	<b>POSTER SESSION II</b>		
16.00	<b>Presentation</b>  topics: 5 and 10  Chairman: D. Maurer  <b>Lecture room 1</b>	<b>Presentation</b>  topics: 3, 7 and 11  Chairman: R. Schaller  <b>Lecture room 2</b>	<b>Presentation</b>  topics: 13 and 14  Chairman: E. Drescher  <b>Lecture room 3</b>
17.30	<b>VISIT TO POSTERS</b>		
18.30	(Topics 3, 5, 7, 10, 11, 13 and 14)		

20.00

Conference Dinner

In an ancient Villa in the Appia Antica area.



# THURSDAY September 9th 1993

	<b>12 - GENERAL ASPECTS OF ANELASTICITY AND MECHANICAL PROPERTIES</b>  Chairman: R. De Baust  <b>Lecture Room 1</b>	<b>14 - EXPERIMENTAL TECHNIQUES NON DESTRUCTIVE TESTING AND INDUSTRIAL APPLICATIONS</b>  Chairman: I. Ritchie  <b>Lecture Room 2</b>
9.00	A.V. Granato* O12-1  The diaelastic effect in thermodynamic properties of condensed matter	W.H. Robinson O14-1  Recent applications of high-damping hysteretic devices for the seismic isolation of buildings, bridges and industrial equipment
9.25	F. Povolo O12-2  Influence of a distribution of relaxation times in the damping of materials	E. Drescher-Krasicka* O14-2  Scanning acoustic imaging of residual stresses in ceramics
9.50	G. Cannelli O12-3  Acoustic emission and self-organized criticality associated with fracture processes during hydrogen precipitation in Nb	M. Koiwa* O14-3  Determination of elastic constants of single crystals by the rectangular parallelepiped resonance method
10.15	Coffee break	
	<b>11 - GRAIN BOUNDARIES, DOMAIN WALLS, INTERFACES</b>  Chairman: W. Benoit  <b>Lecture Room 1</b>	<b>14-EXPERIMENTAL TECHNIQUES NON DESTRUCTIVE TESTING AND INDUSTRIAL APPLICATIONS</b>  Chairman: J. de Fouquet  <b>Lecture Room 2</b>
10.45	Y.M. Soifer* O11-1  Elastic and dissipative properties of metals with ultrafine grain structure	W. Arnold O14-4  Measurements of internal friction in polycrystalline materials using laser ultrasound
11.10	B. Cao* O11-2  Internal friction associated with grain boundaries in Ni-Cr alloys	A. Migliori* O14-5  Resonant ultrasound techniques for measurements of the elastic moduli of solids
11.35	S. Kiss O11-3  The study of internal friction in metal-metal layered materials	B. Augustyniak O14-6  Internal stress evaluation from mechanical Barkhausen effect
12.00	S. Okuda O11-4  Anelasticity of ultra-fine grained polycrystalline gold	B.C. De Cooman O14-7  N-ageing in aluminised steel sheet: an industrial application of high frequency internal friction measurements to point defect/interface interactions
12.25	Lunch	

	<b>9 - THIN FILMS</b>  Chairman: T.S. Kê  <b>Lecture Room 1</b>	<b>14-EXPERIMENTAL TECHNIQUES NON DESTRUCTIVE TESTING AND INDUSTRIAL APPLICATIONS</b>  Chairman: M. Koiwa  <b>Lecture Room 2</b>
14.00	<i>M. Wuttig*</i> O9-4  Internal friction in membranes and thin films	<i>S. Sgobba</i> O14-8  Analysis of the anelastic creep of Al and two Al-Cu alloys
	<b>11 - GRAIN BOUNDARIES. DOMAIN WALLS. INTERFACES</b>  Chairman: T.S. Kê  <b>Lecture Room 1</b>	<b>6 - AMORPHOUS MATERIALS</b>  Chairman: M. Koiwa  <b>Lecture Room 2</b>
14.25	<i>E. Bonetti</i> O11-5  Anelastic relaxation structural stability and trasformation in nanocrystalline metals and alloys	<i>L. Murawski</i> O6-5  Correlation between mechanical and electrical loss in transition metal oxide glasses
14.50	<i>X.S. Guan</i> O11-6  Nonlinear mechanical relaxation associated with the viscous sliding of grain boundaries	<i>V.A. Khonik</i> O6-6  Dislocation-like relaxations in cold deformed metallic glasses
15.15	<b>CONCLUDING REMARKS</b>  <b>Lecture Room 1</b>	

### Topics

1. Point defects.
2. Dislocations and their interaction with lattice imperfections.
3. Phase transformations: a) displacive transitions; b) order-disorder transitions and precipitation.
4. Hydrogen in metals and semiconductors.
5. Oxide superconductors and fullerenes.
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13. Magneto-elastic interactions.
14. Experimental techniques, non-destructive testing, industrial applications.

N.B. Only the authors presenting the contribution have been reported in the Programme.

## PLENARY TALK

- PL-1** 40 YEARS OF DISLOCATION INTERPRETATION OF THE BORDONI RELAXATION - SOLVED AND UNSOLVED PROBLEMS

A. Seeger

## ORAL CONTRIBUTIONS

### 1-POINT DEFECTS

- O1-1** INTERNAL FRICTION AND SOUND VELOCITY OF POLYCRYSTALS AT VERY LOW TEMPERATURES

R. König, P. Esquinazi, D. Valentin, F. Pobell

- O1-2** MECHANICAL LOSS STUDIES OF OXYGEN-OXYGEN INTERACTION IN TANTALUM

G. Haneczok, M. Weller, J. Diehl

- O1-3** ANELASTIC BEHAVIOUR IN Nb-Ti ALLOYS CONTAINING INTERSTITIAL ELEMENTS

O. Florêncio, W.J. Botta F., C.R. Grandini, H. Tejima, J.A.R. Jordão

- O1-4** MIGRATION MECHANISM OF SELF-INTERSTITIAL ATOM IN Mo AFTER LOW-TEMPERATURE IRRADIATION: I-RELAXATION PEAK

H. Tanimoto, H. Mizubayashi, N. Teramae, S. Okuda

- O1-5** INTERSTITIAL SITE OCCUPANCIES AND HYDROGEN TRANSITION PROBABILITIES IN FCC BINARY ALLOYS: STATISTICAL MODEL

A. Biscarini, B. Coluzzi, F.M. Mazzolai

### 2-DISLOCATIONS AND THEIR INTERACTION WITH LATTICE IMPERFECTIONS

- O2-1** DISLOCATION TUNNELING THROUGH A PINNING-POINT IN ALUMINUM

T. Kosugi, D. McKay, A.V. Granato

- O2-2** BORDONI AND HYDROGEN SNOEK-KÖSTER RELAXATION IN FCC METALS

W. Ulfert and A. Seeger

- O2-3** THE NATURE OF DISLOCATION RELAXATION PEAKS IN INTERNAL FRICTION OF SOLIDS

V.P. Kisel

- O2-4** SELF-ORGANIZED CRITICALITY AND DISLOCATION DAMPING

F. Marchesoni

- O2-5** HIGH TEMPERATURE INTERNAL FRICTION IN ALUMINIUM STUDIED BY ISOTHERMAL MECHANICAL SPECTROMETRY

A. Rivière, J. Woignard

- O2-6** THE AMPLITUDE-DEPENDENT INTERNAL FRICTION AND DISLOCATION INELASTIC STRAIN IN ZINC AND ALUMINIUM SINGLE CRYSTALS

S.B. Kustov, S.N. Golyandin

- O2-7** INTERNAL FRICTION DUE TO TWINNING DISLOCATIONS IN Cu-Ge SOLID SOLUTION ALLOYS

H. Numakura, K. Hasegawa, M. Koiwa, H. Fukuda, R. Taniguchi and S. Tsukui

- O2-8** NONLINEAR DAMPING AND HARMONIC GENERATION IN IRON

V.F. Coronel, D.N. Beshers

- O2-9** INTERNAL FRICTION AND DEFECT OF MODULUS IN  $\alpha$ -Fe BASED HIGH ALLOYED (Cr,Mo) HIGH ALLOYS

I.S. Golovin

**02-10 AMPLITUDE-DEPENDENT INTERNAL FRICTION AND PLASTIC PROPERTIES OF CRYSTALS**

S.P. Nikanorov, B.K. Kardashev, S.B. Kustov, A.B. Lebedev

**3-PHASE TRANSFORMATIONS: A) DISPLACIVE TRANSITIONS; B) ORDER-DISORDER TRANSITIONS AND PRECIPITATION**

**03-1 INVESTIGATIONS OF THE ELASTIC BEHAVIOUR IN THE VICINITY OF FERROELECTRIC PHASE TRANSITIONS**

G. Sorge, U. Straube

**03-2 INTERNAL FRICTION OF Fe-Mn-Si ALLOY**

I. Yoshida, H. Ohtsuka

**03-3 THE LOCAL PHASE TRANSITIONS ON MOBILE DISLOCATIONS IN CRYSTALS**

A.L. Korzhenevskii, D.A. Lisachenko

**03-4 STUDIES ON PRE-BAINITIC TRANSFORMATION IN Cu-Zn-Al-Mn ALLOY**

Shen Huimin, Zhang Zhufang, Yang Yanqing, Rui Limin, Wang Yening, Shen Guangjun

**4-HYDROGEN IN METALS AND SEMICONDUCTORS**

**04-1 MECHANICAL SPECTROSCOPY WITH HYDROGEN IN INTERMETALLIC PHASES**

H.R. Sinning

**04-2 REORIENTATION OF DIATOMIC H-Y COMPLEXES IN DILUTED Pd/Y ALLOYS**

G. Hauptmann, M. Ege, W. Ulfert, H. Kronmüller

**04-3 FOUR-SITE TUNNELLING OF H TRAPPED BY SUBSTITUTIONAL Zr IN Nb**

G. Cannelli, R. Cantelli, F. Cordero and F. Trequattrini

**5-OXIDE SUPERCONDUCTORS AND FULLERENES**

**05-1 OXYGEN DIFFUSION AND PHASE TRANSFORMATIONS IN  $R\text{Ba}_2\text{Cu}_3\text{O}_{6+x}$**

G. Cannelli, R. Cantelli, F. Cordero, N. Piraccini and F. Trequattrini

**05-2 LOW TEMPERATURE TORSION PENDULUM MEASUREMENTS ON YBCO SUPERCONDUCTORS**

P. Devos, R. De Batist, J. Cornelis, F. Servaes

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**PL-1**  
**40 YEARS OF DISLOCATION INTERPRETATION OF THE BORDONI**  
**RELAXATION - SOLVED AND UNSOLVED PROBLEMS**

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The August 1949 issue of "La Ricerca Scientifica" contained the 12 page paper "Teoria della dissipazione elastica nei monocristalli secondo la meccanica quantistica: un nuovo effetto di rilassamento" by Piero Giorgio Bordoni of the Istituto Nazionale di Ultracustica "O.M. Corbino" in Rome. From the present point of view the paper is highly remarkable in three respects. (i) It showed that after room-temperature cold-work the fcc metals Cu, Ag, Al, and Pb exhibit at low temperatures a pronounced maximum of the internal friction (in Cu at 90 K when the measuring frequency was  $f = 30$  kHz), in striking conflict with the then textbook wisdom that below room temperature the internal friction of metals decreased continuously with decreasing temperature. (ii) It attempted a dislocation interpretation at the time when there was no direct evidence for the existence of dislocations and the great majority of metallurgists did not believe in them. (iii) The paper was submitted on June 19th 1949, which compares rather favourably with the present-day publication periods.

In 1953 the present speaker's attention to Bordoni's work was drawn by Professor H.-O. Kneser, who had been informed about it by Professor A. Giacomini, then the director of the Corbino Institute. Although I was pleased to see Bordoni's attempt to explain the observed phenomenon in terms of dislocations, I had to answer in the negative Professor Kneser's question whether I believed the author's explanation. This was no long after we had discovered the solitonic properties of kinks. I had realized that the generation of kink pairs (originally called double kinks) in dislocations running approximately parallel to Peierls valleys should give rise to relaxation processes and was looking for such a process. In discussions with E. Kröner it became clear that the relaxation phenomenon observed by Bordoni might be the searched-for process. Since at the time I was busy in writing the chapter on crystal imperfections in the Encyclopedia of Physics, I did not publish this explanation in a separate paper but put it in the Encyclopedia, where it appeared in print in 1955. Although many other explanations were proposed (among them one by W.P. Mason at about the same time as the kink pair explanation), the kink-pair interpretation is the only one which has stood the test of time. One difficulty of the theory, namely that in metals different Peierls barriers exist and that therefore several kink-pair relaxation mechanisms should be observed, was soon removed by the discovery of a second relaxation maximum at lower temperatures by Niblett and Wilks.

The kink-pair generation mechanism accounts for the main properties of the Bordoni and Niblett-Wilks maxima (as well as for a third internal-friction maximum at 11 K [ $f = 53$  kHz] recently discovered by Kosugi and Kino in cold-worked high purity Al) in a straightforward manner. Quite a number of secondary features, however, require rather refined explanations, the quantitative development of which is still going on (cf. the conference paper by Ulfert and Seeger). In spite of substantial recent progress there are a number of questions for which a quantitative theory is still missing. Perhaps the most important of these are problems related to tunnelling processes. Since the kink mass is at most of the order of magnitude of a few hydrogen masses, tunnelling will become important at low temperatures. Arguments will be presented that this should be taken into account when discussing the apparent discrepancy between the low-temperature flow stress of high purity fcc metals and the Peierls stress for dislocations parallel to  $\langle 100 \rangle$  directions as deduced from the Bordoni relaxation.

**O1-1**  
**INTERNAL FRICTION AND SOUND VELOCITY OF POLYCRYSTALS AT VERY LOW TEMPERATURES**

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With the vibrating-wire technique we have extended our investigations of the sound velocity and of the internal friction of polycrystalline NbTi, Ta and Ag to measurements of these acoustic properties on Nb, Nb with Cu-matrix, Al, Pt and further samples of NbTi. The temperature and frequency ranges of our measurements were  $0.04 \text{ mK} \leq T \leq 1 \text{ K}$  and  $0.3 \text{ kHz} \leq \nu \leq 8 \text{ kHz}$ . The polycrystalline samples NbTi, Ta, Nb and Al in the superconducting state as well as normal conducting Pt show quantitatively the same behavior with similar strain dependence as it is observed for amorphous, dielectric materials. When the acoustic intensity is of the order of the thermal energy non-linear effects are observed. In particular, in the non-linear regime ( $T < 50 \text{ mK}$ ) and after a rapid change of the sample temperature we have found that the sound velocity depends logarithmic on time. The glass-like anomalies can be understood assuming the existence of tunneling systems with a broad spectrum of energy splitting and relaxation rates.

**O1-2**  
**MECHANICAL LOSS STUDIES OF OXYGEN-OXYGEN INTERACTION IN TANTALUM**

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Mechanical loss measurements of tantalum doped with various amounts of oxygen (300 to 600 at.ppm) were carried out in the Hz and kHz-range. The experimental data obtained in this way show the following: (i) The mean activation enthalpy calculated from the frequency shift of the Snoek peak does not depend on oxygen content and is  $H_{\text{S}} = 1.10 \text{ eV}$ . (ii) The peak temperature of the Snoek maximum shifts slightly towards higher temperature with increasing oxygen content. (iii) The Snoek maxima are broadened and this broadening increases with oxygen content.

These facts indicate that the interaction between oxygen atoms dissolved in Ta is long range in nature and can be described in the frame of the random cooperative strain interaction (RCSI) model, as it was demonstrated recently for the system Nb-O [1,2]. The analysis of the experimental data by applying the RCSI model gives the following results: (i) The activation enthalpy of the oxygen Snoek relaxation in tantalum is  $H = 1.09 \text{ eV}$  which is a good agreement with  $H_{\text{S}}$ . (ii) The critical temperature  $T_c$  characterizing the oxygen-oxygen interaction in Ta is proportional to the square root of the oxygen concentration and varies from 70 K for 320 at.ppm up to 260 K for 6000 at.ppm.

- [1] M. Weller, G. Haneczok, J. Diehl, phys.stat.sol.(b) 172, 145 (1992).
- [2] G. Haneczok, M. Weller, J. Diehl, phys.stat.sol.(b) 172, 557 (1992).

### O1-3

## ANELASTIC BEHAVIOUR IN Nb-Ti ALLOYS CONTAINING INTERSTITIAL ELEMENTS

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Metals containing solute atoms dissolved interstitially often show anelastic behaviour due to a process known as stress-induced ordering. One manifestation of this anelastic behaviour is the internal friction, which was originally observed by Snoek. The anelasticity has its cause in the stress-induced migration of interstitial atoms in octahedral positions of bcc lattices.

Internal friction measurements as a function of temperature have been performed in a Nb-48wt% Ti heat treated under different conditions. These values were obtained in the temperature range between 250K and 700K, using a torsion pendulum of the inverted K<sub>2</sub>-type, applying a heating rate of 1K/min, at a pressure near 10<sup>-3</sup> bar and oscillation frequency of about 3.5Hz. These data made it possible to obtain spectra of multiple anelastic relaxation as a function of temperature which were resolved into elemental interactions. The following metal-interstitial interactions were identified: Nb-O (430K), Nb-N (541K), Ti-O (467K), Ti-O-O (495K), Nb-O-O (443K) and Ti-O-C-O (523K). The anelastic relaxation parameters, height of the internal friction peak, peak temperatures and activation energy were calculated for each of the process using Debye's elemental peaks.

### O1-4

## MIGRATION MECHANISM OF SELF-INTERSTITIAL ATOM IN Mo AFTER LOW-TEMPERATURE IRRADIATION: I- RELAXATION PEAK

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The behavior of self-interstitial atom (SIA) in Mo has been investigated by many researchers using various experimental methods and several models have been proposed. However, no models succeed in explaining all the experimental results: Based on the anelastic studies after low-temperature irradiation, two- (2DM) or three-dimensional migration (3DM) of  $\langle 110 \rangle$ -split SIA is proposed. On the other hand, the one-dimensional migration (1DM) of SIA is proposed to account for the characteristic feature of the void lattice formation after heavy irradiation at elevated temperatures. This paper deals with the relaxation peak associated with SIA to clarify the migration mechanism of SIA. Part II will deal with the dislocation pinning associated with free-migration of SIA. Whether  $\langle 110 \rangle$ -SIA can rotate during free-migration or not is the heart of the controversy between 3DM and 2DM or 1DM models. 3DM requires that the relaxation peak due to the rotation of  $\langle 110 \rangle$ -SIA (SIA-peak) should be found large enough to explain the strong anisotropic strain field around  $\langle 110 \rangle$ -SIA estimated from Huang scattering. Very recently, we found that the strength of SIA-peak per unit concentration of Frenkel-pair ( $C_{FP}$ ) tends to increase with decreasing  $C_{FP}$ , suggesting that SIA at low  $C_{FP}$  undergoes 3DM. To pursue this issue further, we performed the internal friction measurements of Mo after low-temperature irradiation for  $C_{FP}$  range from 0.1 to 54 ppm. SIA-peak here is observed at 41 K for 500Hz. The strength of SIA-peak per unit  $C_{FP}$  was found to increase strongly with decreasing  $C_{FP}$  below 10 ppm. The extrapolation to lower  $C_{FP}$  gives the relaxation strength expected from Huang scattering at around 0.01 ppm. This result suggests for  $C_{FP} < 0.01$  ppm, all  $\langle 110 \rangle$ -SIA's undergo 3DM. With increasing  $C_{FP}$ , presumably the fractional ratio of the  $\langle 110 \rangle$ -SIA undergoing 3DM decreases rapidly and the remains migrate without rotation. We surmise that the latter behavior is due to the interaction between SIA's.

O1-5  
INTERSTITIAL SITE OCCUPANCIES AND HYDROGEN TRANSITION PROBABILITIES IN FCC  
BINARY ALLOYS: STATISTICAL MODEL

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Several interstitial sites can be energetically distinguishable in a binary alloy, depending on the composition and the atomic arrangement within the nearest and more distant shells of neighbours, therefore, it is of relevance to know which type of sites are occupied by H and D in binary systems. In the last few years we have calculated the probabilities for octahedral site occupancies and for O - O transitions in random fcc alloys. In view of the complexity of the problem, attention was confined to the lowest energy octahedral sites, which were only distinguished one from the other on the basis of the chemical composition of their first coordination shell of neighbours. In these investigations, the probability of finding an octahedral site of a certain type next to a site of given type have been calculated together with the transition probability factors.

In the present work the calculations are extended to include tetrahedral site occupancies, blocking effects and different atomic arrangements within the first coordination shell of neighbours. Explicit expressions are derived for the probabilities of reorientational transition of the H elastic dipole, which are of major interest for anelastic studies of metal-hydrogen systems.

O2-1  
DISLOCATION TUNNELING THROUGH A PINNING-POINT IN ALUMINUM\*

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Dislocation tunneling is often invoked as the source of anomalous mechanical behavior at low temperatures. But there is little convincing evidence for it. Measurements of the stress amplitude necessary for a given magnitude of decrement as a function of temperature have been found to measure the interaction of a dislocation with a single pinning point in relatively pure materials at low temperatures. Classical rate theory predicts the 2/3 power dependence of stress amplitude on temperature observed. In earlier measurements down to 1.6K, no deviations arising from quantum mechanical tunneling were found in Cu, Al, or Pb. In more recent measurements down to 0.6K, we have found no convincing evidence for dislocation tunneling in either the normal or superconducting state in Al-50 ppmAg, Al-20 ppm Cu, Al 25 ppm Mg, and zone refined (ZR) Al. However, in our most recent measurements on ZR Al in the superconducting state down to 0.2K, we finally find a definite flattening of the temperature dependence, which provides strong evidence for dislocation tunneling through a pinning point.

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O2-2  
BORDONI AND HYDROGEN SNOEK-KÖSTER RELAXATION IN FCC METALS

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It is now generally accepted that the Bordoni relaxation is caused by thermally activated kink-pair formation and kink migration on dislocation lines under the action of external stress. The presence of foreign interstitial atoms may reduce the kink mobility. In the limit of strong kink-interstitial interaction it is determined by the diffusivity of the interstitial atoms. As a consequence, the relaxation is shifted to higher temperatures. This process has been followed experimentally by in-situ loading of cold-worked Pd with hydrogen. These experiments have shown that the hydrogen Snoek-Köster relaxation can be obtained from the Bordoni relaxation in this way and that information on the character of the dislocations involved can be derived in this way.

In spite of the success of the theory, a number of open questions remain, some of which could recently be clarified. The linear kink density in thermal equilibrium,  $\rho_k^{eq}$ , has been recalculated, taking into account so-called geometrical kinks as well as the existence of a Peierls potential of the 2nd kind. A derivation of the Paré condition based on the diffusion theory of the kink-pair formation is given. It is shown that the expression for the relaxation time  $\tau$  derived by Seeger (1981) continues to hold in the presence of geometrical kinks and internal stresses if the dislocation length between unsurmountable obstacles for kinks,  $L$ , is replaced by an effective length,  $L_{eff}$ . In particular, the prediction of an unusual temperature dependence of  $\tau$ , at low temperatures determined by  $\tau \propto (\rho_k^{eq})^{-2}$  and at high temperatures by  $\tau \propto (\rho_k^{eq})^{-1}$ , remains valid. It is shown that this prediction is borne out both by the experimental data on pure Cu and by the above-mentioned experiments on hydrogen-loaded cold-worked Pd.

O2-3  
THE NATURE OF DISLOCATION RELAXATION PEAKS IN INTERNAL FRICTION OF SOLIDS  
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The work deals with the effect of shear stress  $s$ , stress rate  $\dot{s}$  ( $10^{-4}$  to  $10^9$  MPa/sec), temperature ( $T=1,3$  to  $1023$  K), impurity state and concentration (IC), predeformation  $e_0$  on the macrodeformation  $e$  and microdeformation (mean free path  $l$  and mean number  $n$  of mobile dislocations, mean pathlength  $L$  and width  $d$  of slip bands, mean density  $N$  of dislocations per slip band and density  $S$  of slip band sources) in NaCl single crystals. It appears that the conditions  $s_{l,n,L,N,S,d}(\dot{s}, T, e_0, IC) = \text{const } s_0(\dot{s}, T, e_0, IC)$  are fulfilled, where  $s_{l,n,L,N,S,d}$  are the deforming stresses at  $l, n, e, L, N, S, d$  are constant. The same similarity of  $s_i(\dot{s}, T, e_0, IC)$  - dependences (at internal friction microdeformation  $i$  is constant) with  $s(\dot{s}, T, e_0, IC)$ -curves in different crystals demonstrate the common character of the mechanisms of micro- and macrodeformation and work-hardening in solids. The extremely low estimates of the Peierls stress in solids ( $s_p < 10^{-3}$  to  $10^{-1}$  G, G is the shear modulus) imply the new explanation of the Bordoni relaxation. This and other relaxation peaks in internal friction of solids are explained by the conservative and non-conservative motion of cross-slip jogs and kinks through the stress fields of the second phase nanoprecipitates and other matrix defects like it was done in the author's works on the temperature yield stress anomalies in the same crystals.

O2-4  
SELF-ORGANIZED CRITICALITY AND DISLOCATION DAMPING  
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The relaxation dynamics of a dislocation network is modelled as a self-organized critical (SOC) process. We assume that in the presence of either a rather strong tensile stress, or significant lattice interactions, the dislocation network re-arranges itself at or close to a critical state. Adding a small perturbation stress causes self-adjusting depinning cascade events at all length and time scales. Contrary to the classic vibrating-string model, no stress scale separation between hysteresis and resonance loss is allowed in a SOC state. Under very general assumptions (conservation and isotropy), finite values for the low frequency damping are obtained, in agreement with recent experimental observations.

## O2-5

### HIGH TEMPERATURE INTERNAL FRICTION IN ALUMINIUM STUDIED BY ISOTHERMAL MECHANICAL SPECTROMETRY

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Isothermal mechanical spectrometry experiments ( $10^{-4}$  Hz - 100 Hz) were carried out on strain hardening aluminium samples after recovery annealings ranging between 300 K and 700 K.

For lower annealing temperatures (until 480 K) internal friction spectra exhibit only a low frequency background. Above 480 K, a relaxation peak stands out progressively against the background.

This peak corresponds to the traditionnal  $K_e$  peak. Its height first increases for annealings below 580 K and then decreases above that temperature : the relaxation parameters undergo large changes after successive annealings.

The peak is attributed to dislocation segment motion in dislocation pile-ups created by the strain hardening and modified by recovery annealings.

## O2-6

### THE AMPLITUDE-DEPENDENT INTERNAL FRICTION AND DISLOCATION INELASTIC STRAIN IN ZINC AND ALUMINIUM SINGLE CRYSTALS

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The aim of this work was to study mechanisms, controlling ADIF and microplastic behavior in hcp and fcc metals. The direct observations of reversible dislocation inelastic strain in 99.997 Zn single crystals simultaneously with amplitude-dependent internal friction (ADIF) measurements have been performed for compression tests at room temperature for strain amplitudes higher than  $10^{-6}$  at frequencies 0.01-2.0 Hz. The experimental data were analyzed taking into account cyclic dislocations motion over short- and long-range obstacles [1].

The comparison of the dependencies of inelastic strain on stress amplitude and prestrain for Al with those for Zn, oriented for basal slip, confirmed that dislocations interaction controls inelastic strain at low frequencies in ADIF range. The strong evidence for Kressel's-Brown model of dislocation hysteretic motion through the internal stress fields [2] was obtained.

The significant amplitude hysteresis or time dependence of ADIF was found for Zn samples at low strain amplitudes. This phenomenon was attributed to mobile pinning points redistribution in glide planes of mobile dislocations. The experimental study of the shape of this distribution was carried out.

Stress-inelastic strain hysteretic loops (HL) of Granato-Lücke (G-L) type were observed for starting loading cycles at the lowest strain amplitudes in aged Zn samples. After few cycles of loading this G-L type HL transformed to HL, corresponding to unlocalized friction model. The conclusion was made that observation of G-L type HL is due to dislocations tearing through the cloud of mobile pinning points rather than dislocation loops breakaway.

1. S.N. Golyandin, S.B. Kustov. Fiz. Tverd. Tela (Sov. Phys. Sol. State), 1992, 34, N12

2. H. Kressel, N. Brown. Dislocation Dynamics, McGraw Hill, 1968, p. 337.

**O2-7**  
**INTERNAL FRICTION DUE TO TWINNING DISLOCATIONS IN Cu-Ge SOLID SOLUTION ALLOYS**

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Copper-based primary solid solution alloys are known to exhibit deformation twinning at room temperature. When such alloys, e.g., Cu-8 at.% Ge, are subjected to a loading-unloading cycle in a tensile test, a hysteresis loop is observed in the stress-strain curve; the pseudoelastic effect is believed to be due to reversible motion of twin boundaries. We have studied anelastic behaviour of Cu-Ge alloys by low-frequency internal friction measurements. Characteristic internal friction is observed in deformed specimens, which increases linearly with temperature in the range from 80 to 280 K and falls rapidly above 300 K. The dependence of the magnitude of the internal friction on the solute concentration and the degree of deformation is similar to that of the pseudoelastic effect. Therefore, the internal friction is ascribed to local oscillatory motion of twinning dislocations. The internal friction disappears completely by electron irradiation after deformation. This suggests that the origin of the internal friction is the motion of individual dislocations.

**O2-8**  
**NONLINEAR DAMPING AND HARMONIC GENERATION IN IRON**

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We report here data obtained at high strain amplitudes under a saturating magnetic field using the same specimens, both annealed and deformed, for which the amplitude dependence at zero field has been reported previously. The damping data, when presented in Granato-Lücke (GL) plots, show (a) for the annealed specimens two linear segments and (b) for the deformed specimens curves with no significant straight segments. Harmonics of order 2 to 5 were observed. For the annealed specimens, the 3rd harmonic showed a remarkable sharp dip at a strain amplitude about  $1.7 \times 10^{-4}$ , a little below the break in the GL plot. These results will be discussed in terms of the GL breakaway model and the Hikata-Elbaum theory of harmonic generation by vibrating dislocations. The latter, based on bowout without breakaway, predicts that edge and screw dislocations should emit 3rd harmonic with opposite phase, offering a possible explanation of the cancellation. A synthesis of the breakaway and bowout models will be attempted.

INTERNAL FRICTION AND DEFECT OF MODULUS IN  $\alpha$ -Fe BASED HIGH ALLOYED (Cr,Mo) HIDAMETS

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Amplitude and temperature dependent internal friction (ADIF and TDIF) and defect of modulus (ADDM and TDDM) of few ferritic materials ( $\alpha$ -Fe,  $\alpha$ -Fe-Cr,  $\alpha$ -Fe-Mo,  $\alpha$ -Fe-Cr-Mo) were investigated in the range of temperature -190...830°C, deformation  $10^{-7}$ ... $10^{-3}$  and frequency 1Hz-21kHz.

With the help of different mechanical tests, magnetic and structure investigations of the influence of annealing temperature (to 1300°C) of cold worked samples on the structure formation, properties and parameters of IF and DM of ferritic steels was provided. Considering the non-linear character of energy of deformation of ferrite, the analysis of the influence of different types of crystalline structure defects on domain walls movement and magnetomechanical energy losses was carried out.

Possibilities of low-temperature treatment ( $T < T_c$ ) were shown from the point of view of damping. Peculiarities of influence of decomposition in Fe-Cr system on non-elastic properties were investigated. Taking mechanism of appearing of microplastic deformation as the base, the influence of preliminary deformation in the range  $10^{-7}$ ... $10^{-1}$  on ADIF was analysed.

## O2-10

## AMPLITUDE-DEPENDENT INTERNAL FRICTION AND PLASTIC PROPERTIES OF CRYSTALS

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A review of works on combined investigations of micro- and macroplastic behavior of fcc ionic crystals, metals with fcc, bcc and hcp structures at ultrasonic frequencies (approx. 100 kHz) and under quasistatic loading at 4.2-300 K, and at frequencies 0.01-1 Hz is presented. Three types of experiments are discussed: i) low frequency reversible dislocation strain in amplitude-dependent internal friction (ADIF) range [1], ii) temperature dependences of yield stress and micro-yield stress, defined from ADIF [2], iii) measurements of acoustoplastic effect (APE) *in situ* during plastic flow under quasistatic load [3]. The APE is shown to occur only in amplitude-dependent region of damping. The similarity law between the temperature dependences of micro- and macroyield stress is found to hold well for all the data available. It gave the possibility to conclude that the same barriers may control the dislocation mobility both under vibrational stress in ADIF experiments and under the quasistatic one at the onset of the plastic flow.

The study of low-frequency reversible dislocation strain in ADIF range served an intermediate step from quasistatic plasticity and fatigue tests to sonic and ultrasonic study of ADIF. The direct observations of inelastic dislocation strain enabled us to make a decision on the validity of different ADIF models and to draw a conclusion that ADIF in general can be described in terms of basic macroscopic features like strain hardening due to reversible dislocation motion, thermally activated and athermal overcoming of short-range obstacles. Each of these mechanisms seems to be dominant for different frequency, amplitude and temperature ranges.

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**O3-1**  
**INVESTIGATIONS OF THE ELASTIC BEHAVIOUR IN THE VICINITY OF FERROELECTRIC  
PHASE TRANSITIONS**

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Measurements of the complex elastic properties of crystals belong to the traditional and the most important investigations of ferroelectric phase transitions. The temperature dependence of elastic stiffness ( $c_{ij}$ ) and compliance ( $s_{ij}$ ) coefficients, their dependence on external mechanical stresses and fields as well as the acoustic damping are of special interest. A fairly new branch of investigations consists in the determination of nonlinear elastic coefficients and the elastic properties at low frequencies. The experimental methods will be shortly summarized. The discussion of these methods involves the comparability of different methods, the influence of experimental boundary conditions and the applicability of some methods.

Starting from established theories a survey of anomalies of elastic coefficients in the vicinity of ferroelectric phase transitions for proper, pseudoproper, weak and incommensurate modulated ferroelectrics will be given. These results will be compared to experimentally obtained data. New trends in the investigation of elastic properties concern cluster and glass like behaviour in some ferroelectrics and will be considered too.

**O3-2**  
**INTERNAL FRICTION OF Fe-Mn-Si ALLOY**

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Anelastic measurements in the low frequency range have been made on an Fe-based recently developed shape memory alloy, Fe-28Mn-6Si-5Cr. Specimens had been quenched from an austenitic region. By the resistivity measurement martensitic transition temperatures were determined as  $M_s = 283K$ ,  $A_s = 368.7K$ ,  $A_f = 401K$ .

On cooling, two internal friction peaks were observed, one at 275K and the other at 238K. These peaks were associated with a frequency change. On heating the lower temperature peak at 238K was observed again but the higher temperature one at 275K disappeared. On heating further another sharp peak was observed at 405K, which clearly corresponded to the inverse transformation to austenite. This internal friction peak was associated with a frequency drop, not with a frequency minimum as reported on non-ferrous shape memory alloys such as NiTi or CuAlNi. By repetition of cooling and heating cycle the above behaviours were reproduced, but each peak height decreased gradually.

When the specimen was austenitized at 1273K and cooled down slowly, both peaks at 275K and at 238K on cooling disappeared and a very flat low peak centred at 205K was observed. This specimen showed no peak at all on heating from 120K up to the room temperature, but a sharp peak at 400K was observed.

Internal friction peaks observed in Fe-Mn-Si shape memory alloy are considered to correspond to martensitic phase transformation and are similar to those observed in other shape memory alloys, but frequency behaviours are quite different from them. No sharp frequency minimum was observed.

### O3-3

## THE LOCAL PHASE TRANSITIONS ON MOBILE DISLOCATIONS IN CRYSTALS

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Although the importance of defect structures in mechanical and acoustic properties is undoubted and the role of defects in local phase transitions (LPT) is commonly recognised, the martensitic LPT on mobile defects remains quite unexplored.

In our recent works we have found out the general principles of LPT-induced microplasticity and examined several particular cases.

The basic idea is the great change of the line tension of dislocations caused by the nucleating of the new phase on them ("coating"). This "coating" is mobile in contrast to the Cottrell clouds, and the character of motion of "coated" dislocations is changed. The kinetics of the 1-st order LPT on mobile dislocations is now to be considered.

We focus our report on our new results concerning the martensitic LPT-induced dislocation anomalies and kinetics of LPT on mobile defects.

### O3-4

## STUDIES ON PRE-BAINITIC TRANSFORMATION IN Cu-Zn-Al-Mn ALLOY

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The behavior of the pre-bainitic transformation in the Cu-Zn-Al-Mn alloy was investigated by using internal friction( $Q^{-1}$ ) measurements and TEM. The results show that there always exists an internal friction peak near 190°C at 1.4 Hz showing the characteristic of the relaxation peak with activation energy of 1.23 eV. The half width of the peak is roughly 1.2 times broader than that of a Debye peak calculated for a single relaxation time. The peak is believed to be associated with the segregation of solute atoms before the formation of orthorhombic 9R bainite, which was confirmed by the composition measurement and the high-temperature transmission electron microscope (TEM) observation, and then the 9R bainite nucleates martensitically in depleted regions of solute atoms in the B<sub>2</sub> phase. The transformation processes mentioned above were also confirmed in isothermal internal friction and TEM experiments.

O4-1  
MECHANICAL SPECTROSCOPY WITH HYDROGEN IN INTERMETALLIC PHASES  
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After a brief survey of previous work on the hydrogen reorientation relaxation in intermetallic phases with amorphous as well as crystalline structures - including own experimental ( $\text{CoZr}_2$ ,  $\text{NiZr}_2$ ,  $\text{NiTi}_2$ ) and theoretical (C16 phases) results - some examples are shown which demonstrate the specific sensitivity of this type of relaxation as a probe to study details of the host materials.

In nanocrystalline materials prepared by crystallization of metallic glasses, the local structure of the grain boundaries can be compared with that of the original amorphous phase by means of the H-induced relaxation parameters. The general result is that these two structures are indeed very similar. However, there are some differences in the H concentration dependence of the respective damping peaks in  $\text{CoZr}_2$  possibly indicating that the degree of disorder is slightly larger in the grain boundaries than in the amorphous phase.

Again in  $\text{CoZr}_2$ , it is shown that the structure of the amorphous phase contains local correspondences with two crystalline phases of the same composition (the metastable  $\text{E9}_3/\text{NiTi}_2$ -type and the stable C16/ $\text{CuAl}_2$ -type phases), depending on whether the probe is located within a pure Zr neighbourhood at low or closer to the Co atoms at higher H concentrations.

In addition to these examples showing the suitability of "mechanical hydrogen spectroscopy" as a local probe with atomic-scale sensitivity, the method may also be used on a more empirical level for characterizing microstructures and phase transformations.

O4-2  
REORIENTATION OF BIATOMIC H-Y COMPLEXES IN DILUTED Pd/Y ALLOYS

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The reorientation of biatomic H-Y complexes in hydrogen-doped diluted  $\text{Pd}_{100-x}\text{Y}_x$  alloys ( $x=1, 2, 4.5, 8$ ) has been studied by means of internal friction in an inverted torsion pendulum and in a vibrating reed apparatus.

As reported earlier [1] the IF of H-doped diluted  $\text{Pd}_{100-x}\text{Y}_x$  alloys shows three maxima that are identified as Zener peak, reorientation peak of biatomic H-Y complexes and Snoek-Köster peak. The width of the reorientation peak exceeds that of a single Debye maximum and increases with the Y content as well as with the H content of the sample. For a fixed H content the maximum shifts to higher temperatures if the Y content of the alloys increases, whereas for fixed Y content there is a shift to lower temperatures caused by increasing H content.

The measured reorientation peak can be fitted by a superposition of Debye maxima using a constant pre-exponential factor and spectra of activation enthalpies for the relaxation times. The analysis of the H-content dependence of the relaxation strength yields a value of  $|\lambda_1 - \lambda_2| = 0.036$  for the elastic anisotropy of the biatomic H-Y complexes. The activation parameters for the relaxation time of the reorientation of an isolated complex are 0.18 eV and  $3 \cdot 10^{-12}$  s.

The properties of the activation-enthalpy spectra can be interpreted in terms of a model taking into account the elastic long-range interaction between Y atoms in the vicinity of an H-Y complex and the complex itself.

[1] G.Hauptmann, W.Ulfert and H.Kronmüller, Materials Science Forum, 1993, Vol.119-121, 109

## FOUR-SITE TUNNELLING OF H TRAPPED BY SUBSTITUTIONAL Zr IN Nb

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We measured the elastic energy dissipation of  $\text{NbZr}_{0.0045}\text{H}_x$  single crystals excited with extensional and torsional vibrations along their  $[100]$  and  $[111]$  directions. The dissipation below 40 K is due to the redistribution of H trapped by Zr within tunnelling states, and consists of two components. One is a small peak near 2 K whose height immediately saturates with the H concentration and which appears only under  $[100]$  extensional and  $[111]$  torsional vibrations. The major component extends up to 40 K and has a similar but less marked dependence on the vibration type. Such a dependence of the relaxation strength on the applied stress allows to distinguish between different tunnelling geometries. It is shown that it is exactly as expected from four-site tunnelling near the substitutional impurity, whereas two-site tunnelling can hardly explain it.

05-1  
OXYGEN DIFFUSION AND PHASE TRANSFORMATIONS IN  $\text{R}\text{Ba}_2\text{Cu}_3\text{O}_{6+x}$

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The most relevant results from anelasticity experiments on  $\text{R}\text{Ba}_2\text{Cu}_3\text{O}_{6+x}$  are reviewed and new results are produced. The dissipation curves of  $\text{R}\text{Ba}_2\text{Cu}_3\text{O}_{6+x}$  present several peaks over the whole temperature scale, both of thermally activated nature and associated with phase transformations. Most of the peaks have been correlated with the various possible motions of the mobile O atoms in the  $\text{CuO}_x$  planes. The processes with higher intensities and activation energies should be due to long jumps involving significant reorientation of the elastic dipole, whereas some of the smaller peaks with lower activation energies are explained in terms of the hopping and ordering of the O atoms within off-centre positions in the Cu-O chains. Emphasis is put on the phase transformations observed around 100-150 K, to which little attention has been devoted up to now. New measurements at the lowest O stoichiometries confirm the hypothesis of an extraordinarily high mobility of the isolated O atoms.

05-2  
LOW TEMPERATURE TORSION PENDULUM MEASUREMENTS ON YBCO  
SUPERCONDUCTORS

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The mechanical damping spectrum observed below room temperature in YBCO superconductors of various stoichiometries and microstructures is characterized by a fairly rich collection of absorption peaks. In spite of intense research efforts, the precise microscopic mechanisms for these peaks have not yet been established beyond doubt.

In the present work, an attempt is made to try correlating some of these below room temperature damping peaks with specific microstructural features of the material, such as oxygen rearrangement, twin boundaries, phase boundaries, etc.

O5-3  
ULTRASONIC ANOMALIES AND PHASE TRANSITIONS IN THE  $Y_{1-x}Ba_2Cu_4O_8$  AND  $Y_{1-x}Ca_xBa_2Cu_4O_8$  SYSTEMS

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Ultrasonic measurements were performed as a function of temperature in single phase  $YBa_2Cu_4O_8$  and in  $Y_{1-x}Ca_xBa_2Cu_4O_8$  ceramic systems. Specific heat measurements were also performed in the latter case. In the pure Y124 material a pronounced dissipation peak accompanied by a velocity increase was observed at about 260 K. We propose it is caused by the formation of an antiferroelectric or ferroelectric phase associated with ordering of oxygen atoms in some off-center positions. Below 200 K a thermal hysteresis develops with no corresponding anomaly in attenuation. The area of the loop seems to be determined by the percentage of pore volume and by grain size. A reversible defect model is proposed to explain this behaviour. In the Ca-substituted material a distinct anomaly due to a phase transition is demonstrated to occur at 150 K in the entire available range of substitution  $x=0.025$  to  $0.1$ , both in sound velocity, attenuation and specific heat. Possible explanations are discussed.

O5-4  
ELASTIC AFTEREFFECT STUDY OF THE FERROELASTIC TETRAGONAL-ORTHORHOMBIC  
PHASE TRANSITION IN  $YBa_2Cu_3O_x$

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The tetragonal-orthorhombic phase transition in  $YBa_2Cu_3O_x$  is the result of a redistribution of oxygen atoms between two differently oriented types of sites in the CuO (or Cu(1)) atomic plane. In the symmetry-breaking orthorhombic phase, the two types of sites are commonly called  $O(1)$  and  $O(5)$  sites, respectively. The redistribution of the oxygen atoms, and thus the phase transition, is stress-sensitive as a consequence of the orthorhombic lattice distortion in the case of a preferential occupation of one of the two types of sites. This means that  $YBa_2Cu_3O_x$  samples exhibit a ferroelastic behavior with an anelastic susceptibility (relaxation strength) that diverges in the neighborhood of the tetragonal-orthorhombic phase transition.

We carried out elastic aftereffect measurements to investigate the ferroelastic behavior of sintered  $YBa_2Cu_3O_x$  samples. The measurements were performed in the temperature range between 100 and 150 °C and with various oxygen concentrations  $x$  ( $6.25 \leq x \leq 6.95$ ). The experiments demonstrated an enormous increase of the elastic susceptibility (or relaxation strength) in the neighborhood of the tetragonal-orthorhombic phase transition at  $x \approx 6.4$ . The time dependence of the anelastic relaxation characterizes the jump rate of the oxygen atoms between the two types of sites in the CuO plane. The measured time dependence can be described by a wide spectrum of relaxation times with activation energies centered around  $\sim 1$  eV. The comparison with literature data for the self diffusion coefficient of the oxygen shows that oxygen jumps between the two differently oriented types of sites in the CuO plane represent the rate determining step in long-range oxygen diffusion.

05-5  
PIEZOELECTRIC EFFECT AND ULTRASONIC ATTENUATION NEAR  $T_c$  IN SINGLE  
CRYSTALLINE  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  SUPERCONDUCTORS

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The attenuation of ultrasonic waves in single crystalline high- $T_c$   $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  has been measured. At  $T_c$  a pronounced peak is observed for the longitudinal  $c_{11}$ -mode. The existence of a piezoeffect in the high- $T_c$  superconductor has been confirmed in the temperature range from 4 to 280 K in a pulse-echo ultrasonic measurement employing an  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  crystal as a transducer for the longitudinal  $c_{33}$ -mode. The peak near  $T_c$  for the absorption of the  $c_{11}$ -mode can be explained by a model taking into account the piezoelectric properties of the material and a treatment of the electron gas according to Drude theory.

05-6  
ULTRASONIC INVESTIGATIONS OF DIELECTRIC PROPERTIES OF  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$

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Amongst its high superconducting transition temperature one of the most surprising findings in  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  is the existence of piezoelectricity even in the superconducting state. Therefore, the inversion symmetry must be destroyed in the 123-compound below a certain temperature. Furthermore, piezoelectricity indicates that a close relationship between lattice distortions and the electric polarizability might exist. Hence, the basic question arises whether or not  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  also becomes ferro-/antiferroelectric because since the early days of superconductivity it is believed that there is a possible link between high- $T_c$  superconductivity and the formation of a spontaneous electric polarization. It is shown that both the elastic properties and the sound-induced electromagnetic fields give clear evidence for the formation of an electrically polarized state above  $T_c$ .

OS-7  
SUPERCONDUCTING ENERGY GAP IN MELT TEXTURED  $\text{YBa}_2\text{Cu}_3\text{O}_7$  FROM LOW FIELD  
ULTRASONIC MEASUREMENTS

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It appears to be possible to determine the superconducting energy gap and its temperature dependence in the domain boundaries of melt textured  $\text{YBa}_2\text{Cu}_3\text{O}_7$  from velocity and attenuation measurements in low magnetic fields. Both attenuation and velocity increase at the penetration field of such a sample. The increase in velocity is proportional to the separation between the vortex lines at this field. The increase in attenuation is produced by a relaxation type of interaction. Quasi-particle relaxation across the superconducting energy gap are produced as the oscillating vortex lines leave behind quasi-particles in the normal core and encompass quasi-particles in the ground state. If a fraction of the relaxation time associated with this process is related to electron-phonon interaction then the temperature dependence of the inverse of the relaxation time yields a BCS superconducting energy gap temperature dependence.

This work was supported by the Office of Naval Research.

06-1  
LOW ENERGY EXCITATIONS IN AMORPHOUS MATERIALS - ACOUSTIC EXPERIMENTS  
AND COMPARISON WITH THEORETICAL MODELS

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At low temperatures, many properties of amorphous solids are dominated by low energy excitations with a wide spectrum of energies below a few Kelvin. These excitations are absent in defect free crystals and they are believed to be caused intrinsically from the irregular structure of the material. It is widely accepted that their origin is the tunneling motion of atoms or small clusters through the barrier between two adjacent potential wells. Depending on the theoretical model, the required broad spectrum of arbitrarily small energy splittings of the thus formed two level systems can have different reasons. According to the tunneling model and the soft potential model the distribution of energies comes from the distribution of parameters describing the double well potentials, whereas in more recent theories a strong phonon-mediated coupling between tunneling systems is invoked. All these models claim to explain the universal thermal properties of amorphous solids, however only the first two of them are developed far enough to predict also dynamical properties, e.g. the acoustic behaviour, which seem to be more decisive.

In this paper, the most important and characteristic predictions of the tunneling model for the temperature dependence of sound attenuation and velocity are reviewed and compared with experimental results. Special attention is given to recent vibrating reed experiments on vitreous silica. Moreover, some results on vapour condensed amorphous films, namely mixtures of noble gases and the amorphous metal AuSn, will be presented. These films were studied with surface acoustic waves at 300 MHz.

06-2  
THEORETICAL BASIS AND GENERAL APPLICABILITY OF THE COUPLING MODEL TO  
RELAXATIONS IN SYSTEMS INVOLVING CORRELATED MOTIONS: AMORPHOUS  
POLYMERS, GLASSY IONIC CONDUCTORS, PRECIPITATES AND COLD WORKED METALS  
(SNOEK-KOSTER RELAXATION)

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The coupling model proposed more than a decade ago for the description of relaxations in complex correlated systems by one of us (KLN) have been shown repeatedly to be applicable to amorphous polymers, viscous liquids, the glassy state, metallic glasses, glassy ionic conductors and etc. In these examples it is rather obvious that the relaxing species are dense packed and mutually interacting. As a consequence, the correlations (i.e. cooperativity) between them have a strong influence on the relaxation dynamics, and that is one of the reasons why the coupling model is successful in describing their relaxations. The applicability of the coupling model to metal physics including precipitates and the Snoek-Koster relaxation is less obvious but has recently been justified and demonstrated to be relevant by one of us (YNW). In this presentation we shall discuss the theoretical basis of the coupling model, and show very recent neutron scattering experiments and molecular dynamics that have provided direct evidence to support it. A few examples of applications of the coupling model to problems that we believe are of interest to participants of ICIFUAS-10 will be elaborated. Additional experimental data and analyses will be presented to support our contention.

### O6-3 EFFECT OF PASSING ELECTRIC CURRENT ON THE ELASTIC PROPERTIES OF AMORPHOUS ALLOYS

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Recently for various amorphous alloys,  $\text{Cu}_{50}\text{Ti}_{50}$ ,  $\text{Cu}_{50}\text{Zr}_{50}$  and  $(\text{Cu}_{30}\text{Zr}_{70})_{92.5}\text{Al}_{7.5}$  (a-CuTi, a-CuZr and a-CuZrAl, hereafter), we found that both the structural relaxation (SR) and crystallization can be enhanced under passing electric current (PEC) of current density,  $i_d$ , of about  $10^3 \text{ A/cm}^2$ , and that the observed effect on SR suggests an enhancement of electromigration through some collective motion involving many atoms. Apart from the effect of PEC, recent diffusion studies also report a collective motion of many atoms in a-alloys. At low temperatures below SR, for the elastic property of a-CuTi and a-CuZr for the strain amplitude  $\epsilon_t < 3 \times 10^{-3}$ , we found that with increasing  $\epsilon_t$ , the resonant flexural-vibration frequency,  $f$ , of the reed-specimens increases showing a saturation, suggesting that the constituent inelastic strain,  $\epsilon_i$ , shows an increase which is followed by a saturation. This reversible inelastic behavior can be explained as the atomic-bond-orientational-anisotropy (ABOA) under stress proposed by Egami. These facts remind us that the reversible ABOA may be expected under PEC at low temperatures below SR.

In the present paper, we investigated the effect of PEC on  $f$  and the internal friction,  $Q^{-1}$ , for a-alloys, mainly a-CuZrAl, by means of the vibrating-reed technique. To remove joule heat due to PEC, the measurements were made in  $10^6 \text{ PaHe}$ . The specimen temperature showed, however, a small increase under PEC, which was separately calibrated. It is noted that after the calibration, both  $f$  and  $Q^{-1}$  remain unchanged for crystalline metals in the present range of  $i_d \leq 5 \times 10^3 \text{ A/cm}^2$ , and in contrast, they show a clear change in a-alloys: For a-CuZrAl at room temperature, with increasing  $i_d$ ,  $f$  increases and  $Q^{-1}$  decreases slightly for low  $i_d$  and almost linearly for  $i_d$  beyond about  $2 \times 10^3 \text{ A/cm}^2$ , e.g.,  $f$  increases by 2% and  $Q^{-1}$  decreases by 40% at  $i_d = 5 \times 10^3 \text{ A/cm}^2$ , respectively. Both the  $f$  vs.  $i_d$  and the  $Q^{-1}$  vs.  $i_d$  data remain unchanged for the repetition of measurements in the present  $i_d$  range. The very similar results are also observed at 80K, suggesting that the atomic processes responsible for the changes in  $f$  and  $Q^{-1}$  under PEC is athermal.

### O6-4 ANELASTICITY AND VISCOSITY OF SUPERIONIC CONDUCTING GLASSES

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The dynamical mechanical properties of  $(\text{AgI})_x(\text{AgPO}_3)_{1-x}$  ( $x = 0 - 0.5$ ) superionic conducting glasses have been investigated from the room temperature to the glass transition temperatures ( $T_g = 360 - 420 \text{ K}$ ). The viscoelastic behavior of the material was especially noted, and was studied by shearing a specimen and observing the time dependence of the shear strain using an optical method. The principle of the measurement is as follows. The lower and upper faces of a cubic specimen are bonded to fixed and movable testing plates, and a constant shearing load is applied to the upper plate. The lateral displacement of the plate is recorded by using the HP-5528A laser measurement system utilizing the optical heterodyne interferometric method. The sensitivity of the displacement measurement is 10 nm. It can be shown that the mode of deformation of the specimen is nearly pure shear.

The time dependence of the shear strain of the specimen was observed under constant shearing load (stage I), and then after the load was removed (stage II). In stage II, the anelasticity (mechanical relaxation) is observed. In stage I, the viscosity (irreversible time-dependent deformation) is accompanied with the anelasticity. The experiments were performed for specimens with different compositions at various temperatures. The data were analyzed by adopting a mechanical model of standard linear solid + viscous solid; and the viscosity, the anelastic relaxation time, and the anelastic modulus were determined. The viscosity and the anelastic modulus decrease slowly and then rapidly as the temperature is increased up to the glass transition. The temperature dependences are systematically altered when the specimen composition  $x$  is gradually changed. The anelastic relaxation time is, however, almost independent of temperature for all kinds of specimens. The relation between the present experimental results and the superionic conductivity of the material is now being considered.

#### O6-5

### CORRELATION BETWEEN MECHANICAL AND ELECTRICAL LOSS IN TRANSITION METAL OXIDE GLASSES

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Internal friction and dielectric loss measurements have been performed on oxide iron phosphate glasses.

The internal friction in iron phosphate glasses containing different glass modifiers has been studied in the temperature range from 120 K to 700 K using low frequency torsion pendulum. The dielectric loss were investigated by two independent methods: the absorption current method and AC transformer bridge.

The internal friction spectra exhibit the low temperature peak that can be related to the electron transfer between iron in different valency states. It is confirmed that the dielectric loss peak and DC conductivity revealed a similar activation energy as internal friction peak.

The obtained data have been interpreted in the framework of a new model of relaxation in glasses that has been recently proposed by Hunt [A. Hunt, *J. Non-Cryst. Solids*, **144** (1992) 21]. Its basic concept is the transition of relaxation process from local (parallel) at high frequencies through semi-local at intermediate frequencies to strongly non-local at low frequencies. This theory applied to internal friction and electrical loss data yields to consistent process parameters.

#### O6-6

### DISLOCATION-LIKE RELAXATIONS IN COLD DEFORMED METALLIC GLASSES

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Inhomogeneous (localized) deformation of metallic glasses (MG) results in a number of specific anomalies of low temperature ( $30 < T < 300$  K) anelastic properties which are almost identical to that in predeformed crystalline metals.

Internal friction (IF) temperature dependences of as-cast MG have no peculiarities. The main feature of anelastic behaviour of cold-worked MG consists in the presence of large internal friction peak in the range  $220 \leq T_{\text{peak}} \leq 280$  K (at frequency  $\approx 300$  Hz). The IF peak is observed only in weakly predeformed samples (by 2+4 %), disappears after large predeforming (by 5+15 %) and can be completely suppressed by electron irradiation. In the as-cast state IF is amplitude independent. Predeforming results in strong amplitude dependence and at  $T < 150+180$  K IF is mainly hysteresis. Homogeneous viscous deformation has no effect on low temperature IF.

It is concluded that inhomogeneous deformation results in appearance of new, non-characteristic for as-cast state dislocation-like defects which determine the observed anelastic anomalies.

## O7-1

### ANELASTIC BEHAVIOR OF BARIUM TITANATE BASED CERAMIC MATERIALS

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The internal friction ( $Q^{-1}$ ) and Young's modulus ( $E$ ) of  $\text{BaTiO}_3$ -based ceramics were measured versus temperature from 170 K to 420 K. Rectangular bars of the materials were driven electrostatically in flexural vibration at a resonance frequency of about 3 kHz. The curves of  $Q^{-1}(T)$  and  $E(T)$  allow the study of the three following phase transformations: rhombohedral to orthorhombic (about 200 K), orthorhombic to tetragonal (about 270 K), and tetragonal to cubic (about 395 K). Only the first three phases are ferroelectric, the cubic phase is paraelectric.

The curves of  $Q^{-1}(T)$  and  $E(T)$  have been obtained on non-doped ceramic and on ceramics doped with several concentrations of niobium and cobalt. These same dopants are currently used for the manufacture of capacitors, in order to achieve low dielectric loss and to limit the variation of permittivity in the temperature range  $-55^\circ\text{C}$  to  $+125^\circ\text{C}$ . These dopants inhibit grain growth during sintering, and the intragranular diffusion of the dopants is limited to the periphery of the grains. The resulting heterogeneous microstructure, called the "core-shell" structure, leads to a distribution of the phase transition temperatures. Thus, the variations of  $E$  and  $Q^{-1}$  are considerably attenuated by the presence of the dopants. The influence of the polarization on the variation of internal friction with temperature is also studied. The appreciable  $Q^{-1}$  in the ferroelectric phases is associated with domain wall motion; the internal friction is substantially less in the paraelectric phase.

## O7-2

### INTERNAL FRICTION IN CERIA CERAMICS DOPED WITH ALKALI EARTH METAL OXIDES

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Ceria and 5 mol% of alkali earth metal oxide powder were mixed in EtOH by ball milling and the mixed powder was fired at 1273 K for 3 hours in order to make solid solution of ceria and alkali earth metal oxide. The calcine powder was crashed by a mortar. And then, the crashed calcine powder were fired to make specimens at 1673 K for 6 hours in an air. Measurements were made on internal friction and electrical conductivity in temperature range of room temperature to 700K. Internal friction and electrical conductivity were measured as a function of temperature and frequency by the flexural vibration and complex impedance method, respectively. Internal friction has minimal value at room temperature and reaches the maximum at about 450K in all samples, with increasing temperature. The intensity and half width of internal friction peaks are affected strongly with the ionic radius of alkali earth metal ion as a dopant. Internal friction peaks have a multiple relaxation time. The peak intensity of ceria ceramics, doped with calcium ion, was the strongest and the half width of that was the largest out of the samples. On the other hand, the peak intensity of ceria doped with magnesium ion was very weak and the half width of that was narrow. Electrical conductivity is related to the internal friction peak intensity. Internal friction and electrical conductivity depended on local strain due to difference of ionic radius between cerium ion and dopant ion. Internal friction peak mechanism and electrical conductivity will be discussed from the point of view of the local strain in the vicinity of the dopant oxygen ion vacancy pair.

**07-3**  
**MECHANICAL AND DIELECTRIC LOSS RELATED TO FERROELECTRICS AND RELAXOR  
FERROELECTRICS PHASE TRANSITION**

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Mechanical and dielectric loss are measured for both KDP and TGS near  $T_c$  in kHz range. A loss peak ( $P_2$ ) appears at several degrees below  $T_c$ , and the peak position moves to higher temperature as frequency increases, but the relaxation time does not obey the Arrhenius relation. Further studies indicate that this peak is associated with domain walls. With taking account of the rapid change of order parameter, domain wall density, the mobility of domain walls and the interaction among the walls with temperature near below  $T_c$ , the  $P_2$  peak can be explained successfully.

Moreover, there is also a similar but broader loss peak at several degrees below  $T_m$  for relaxor ferroelectrics, but the analysis of the dissipation with Debye equation is known to yield erroneous results for both the activation energy and the pre-exponential factor. With taking account of the dynamics of domain walls near  $T_c$  developed above and the correlation among the micro-domain moments, and by using the coupling model proposed by Ngai et al to relaxors, the calculated mechanical and dielectric loss of relaxors are in accordance with the experimental data fairly well.

**07-4**  
**CHANGE OF SIGN OF THE PIEZOELECTRICITY IN  $\text{BaTiO}_3$ -PMMA COMPOSITES WITH  
FREQUENCY AND TEMPERATURE**

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It is still an open question why poled ferroelectric polymer films like PVDF, - contrary to fully crystalline ferroelectrics -, have a strong but negative piezoelectric constant  $d_{33}$ . It has been argued theoretically that the piezoelectricity in only partially crystalline materials originates from a complicated interaction of small lamellar crystals with their amorphous surroundings. Since the pure crystalline phase by itself is not experimentally accessible, these theories, however, are not yet confirmed. Here we present a very clear and simple model-substance for the piezoelectric behaviour of these semicrystalline materials: Small crystallites of  $\text{BaTiO}_3$  being embedded in an amorphous matrix of PMMA. The composite films having a thickness of approx. 30  $\mu$  and containing a volume fraction of 30% of  $\text{BaTiO}_3$  particles were poled at temperatures higher than the Curie-temperature for the ferroelectric phase transition of  $\text{BaTiO}_3$  by an electric field of 45MV/m. The change in thickness of the films when applying an electric field was measured by interferometric means. Though crystalline  $\text{BaTiO}_3$  alone has a positive piezoelectric constant these composite films show a strongly negative piezoelectricity at low frequencies quite similar to PVDF. At higher frequencies ( $> 10$  kHz), however, the sign of the piezoelectric constant changes to become positive. This behaviour and also its measured temperature dependence will be explained by a simple theoretical model. According to this model it should be possible to design a heterogeneous artificial material, that has a piezoelectric constant predetermined sign and magnitude.

## O8-1

### INFLUENCE OF A CHANGING MICROSTRUCTURE ON HIGH TEMPERATURE RELAXATION PEAKS, AN EXAMPLE WC-11wt%Co

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WC-11wt% Co cemented carbide exhibits a relaxation peak at about 950 K in the 1 Hz frequency range. Though the apparent relaxation parameters are unusually high: 2.5 to 7 eV for the activation energy and  $10^{35}$  Hz for the limit attempt frequency. Moreover, the apparent activation energy increases with the temperature and superimposed to the relaxation peak, an extra damping is detected which is due to the heating or cooling rate (T effects).

These phenomena can be interpreted by an evolution of the microstructure during the internal friction measurements. Effectively, taking into account the microstructural modifications, it is possible to modify the classical equation for dislocation damping in order to predict the shape of the associated relaxation peaks. Generalization of this approach can supply a method for analysing the damping peaks which present non classical characteristics.

## O8-2

### IDENTIFICATION AND CHARACTERISATION OF RECLAIMED AND RECYCLED POLYMERS BY ULTRASOUND ATTENUATION

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Ultrasound attenuation analysis has been demonstrated to be a rapid, reliable and cost-effective method of identifying and characterising polymeric materials and components, which can assist in the separation and reclamation of thermoplastics from polymer waste streams. A combination of the broadband ultrasonic attenuation (BUA) and transit time for an ultrasonic pulse, in either pulse-echo or transmission modes, can be combined to attain a signature of the material under investigation. The HL (Hull/Langton) Index is independent of component dimensions, and hence is a powerful tool for the identification of components of complex shape where access to all surfaces may be restricted. We have demonstrated the ability to identify a wide range of polymeric materials, and the level of contamination in reclaimed materials. In house recycling of polymers by material processors is of particular concern due to the possibility of multiple recycling of base materials resulting in increased material contamination, and hence, degradation of component properties.

### O8-3

## DYNAMIC MECHANICAL ANALYSIS OF GLASS TRANSITION PEAKS IN POLYMERS AND COMPOSITES

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Dynamic mechanical analysis (DMA) is currently of considerable interest for investigating transitions and other behavioural changes in polymeric solids and in composites based on them. As part of a study of the effects of the surface treatment of carbon fibres, we have attempted to use the method as means of providing information on structural changes occurring at the interface between fibres and matrix or the associated interphase. In order to attempt a quantitative approach, we first analysed the shape of the glass transition loss peak in the plain epoxy thermoset resin that formed the composite matrix in terms of a power law rather than the conventional Arrhenius or Williams-Landel-Ferry models, and were able to show that the power law was able to describe the loss peak satisfactorily both from the mathematical and philosophical points of view. These preliminary results were presented at ICIFUAS-10.

Since then we have used the same methods to analyse the behaviour of thermoplastic polymers like PMMA and polycarbonate, and have found, quite unexpectedly, that the same model, with almost identical fitting parameters also describes the shape of the  $T_g$  peak in these materials, despite their very different structural character.

We have also analysed the shapes of the main loss peaks in epoxy resins containing carbon fibres surface treated by electro-polymerisation and plasma polymerisation methods in order to modify the interface and, in consequence the load transfer characteristics of the composite. The power law function for the plain resin could be fitted with very little modification to the loss peaks of composites with both treated and untreated fibres, despite the substantial effect of the carbon fibres on the stiffness of the material and despite the fact that the surface treatments brought about marked changes in the interfacial shear strength of the composite material. We are therefore led to doubt claims that DMA is a sensitive method for studying these effects.

### O8-4

## JET: HISTORICAL, PHYSICAL AND MATERIAL ASPECTS

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Jet is a fossil hydrocarbon found in scattered locations throughout the world. It has the chemical composition of a sub-bituminous coal, but is singularly free of mineral inclusions common in coals. Consequently it does not weather in a moist atmosphere. For this reason, and because it carves well and takes a high polish, it is useful in the making of jewelry. Indeed, it has been used for jewelry at least as long as amber has been used. Ice-Age relics of jet have been found near Stuttgart, apparently made from material found in the Posidonia Shale of Lias  $\epsilon$ . Old carvings have been found in England and Scotland, largely made from jet found in Yorkshire.

We have secured samples from the Holzmaden shale, from the Yorkshire mines and from the desert SW of the USA. Using internal friction measurements in the Hz range, we have determined that each jet has a characteristic mechanical loss spectrum with varying loss maxima  $\alpha$ ,  $\beta$ , and  $\gamma$ . Thus they differ greatly in "polymeric" character, even though they have nearly the same overall chemical composition. The relaxation parameters of the  $\beta$ -maximum were determined from low frequency forced vibration measurements ( $10^{-3}$  Hz - 10 Hz) as  $H = 0.47$  eV,  $\tau_\infty = 1.6 \times 10^{-13}$  s.

## TRANSIENT INTERNAL DAMPING IN ALUMINIUM BASED METAL MATRIX COMPOSITES

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This paper aims at describing the properties of the transient low frequency internal damping (I.D.) that has been observed in various aluminium based microheterogeneous materials : Al-12.8%Si alloy with large spheroidized silicon precipitates, Al reinforced with 12 vol% of SiC particles, 7075Al alloy with 15 vol% of SiC particles.

I.D. has been investigated by measuring the logarithmic decrement of the freely decaying oscillations of a torsion pendulum, in the temperature range 550-50 K.

On cooling the specimen, the I.D. spectrum,  $\delta(T)$ , is characterized by a broad poorly defined maximum, whose magnitude and position, in the range 100-200K, depend on the tested material. Moreover, the I.D. level is increased with increasing the cooling rate or decreasing the frequency or the amplitude of oscillation. Finally, in most cases the I.D. spectra  $\delta(T)$  measured on heating exhibit again a maximum but show a rather different behaviour with respect to cooling : for all microheterogeneous materials the low temperature side of the spectrum is strongly lowered, but conversely the high temperature side is enhanced. Moreover, in the case of the 7075-SiC composite, there is no more maximum on heating.

It is proved that these phenomena are linked with the presence of particles because the unreinforced materials do not exhibit such a phenomenon.

Finally, the phenomena are interpreted qualitatively in terms of emission and movement of dislocations that could be induced, during the I.D. measurement, by the variation of internal stresses around particles due to the thermal expansion mismatch between the aluminium matrix and particles in the various investigated microheterogeneous materials.

**O9-1**  
**GRAIN BOUNDARY SLIDING IN THIN SUBSTRATE-BONDED Al -FILMS**

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The internal friction of thin substrate bonded Al and Al-alloy films has been found to be significantly different from that observed for bulk Al. In particular the activation energy of the underlying relaxation mechanism was found to be that of grain boundary diffusion for the thin films in contrast to that of volume diffusion for the bulk material. Though the technique has already successfully been applied to characterize various aspects of these metal films [1,2], the microscopic mechanisms leading to the anelastic behavior were poorly understood.

We report on a systematic study of the influences of film thickness, grain size, film/substrate interface, dopant atoms in the Al-films, and different stress modes in the substrate on the anelastic relaxation spectrum. It will be concluded that the underlying relaxation mechanism both in bulk and thin-film Al is grain boundary sliding where the apparent difference between the two cases is due the difference in grain size which is about three orders of magnitude.

1. H.G. Bohn and C.M. Su, Materials Science Forum **119-121**, 273 (1993); C.M. Su and H.G. Bohn, Materials Science Forum **119-121**, 285 (1993)
2. H.G. Bohn and C.M. Su, Mat.Res.Soc.Symp.Proc.Vol. **239**, 215 (1992).

**O9-2**  
**LOSS MECHANISMS IN FINE-GRAINED FERROELECTRIC CERAMIC THIN FILMS FOR ULSI MEMORIES (DRAMs)**

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The principal requirements for use of high-dielectric films as capacitors in planarized ultra-large scale integrated circuit DRAMs (dynamic random access memories) are low d.c. leakage current and low dielectric loss tangents. However, with very few exceptions, the loss mechanisms are unknown in the small-grain ceramic ferroelectrics used in these films. In the present work we describe the frequency dependences of the two materials favored at present, PZT (lead zirconate-titanate) and BST (barium strontium titanate) for 50-100 nm diameter sol-gel preparations at frequencies up to 3 GHz. Loss tangents of 0.001 to 0.014 are observed. Limitations on use of such devices for higher frequency (50 GHz) phased-array radar are discussed.

### O9-3

#### INTERNAL FRICTION AND ELASTICITY OF V AND Cr FILMS ON Si-SUBSTRATES

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The internal friction of V and Cr films on Si-substrates has been investigated over a temperature range between 100 K and 800 K with a measuring frequency of about 1 kHz. Films of  $0.5 - 1 \mu\text{m}$  thickness were deposited on substrates ( $70 \times 7 \times 0.5 \text{ mm}^3$ ) cut from a commercial Si-wafer. The internal friction was measured in the flexural free-free bar mode. Although the background of the internal friction is less than  $10^{-5}$  below 150 K, it increases with the temperature and takes a typical value of  $1.5 \times 10^{-4}$  at 500 K. The background internal friction is explained in terms of the thermoelastic damping of Si. For a Cr-deposited specimen, no detectable change can be observed in the internal friction spectrum. However, the frequency vs. temperature curve shows a shallow dip at the Néel temperature of Cr (310 K). The dip becomes clearer after annealing at 873 K. For an as V-deposited specimen, an oxygen Snoek peak is observed at 570 K, but it decreases gradually by the in-situ annealing at higher temperatures. This behaviour of the Snoek peak is different from that observed for bulk V in which the oxygen Snoek peak increases with annealing. This feature may be explained as follows: An oxide formation takes place over the volume for the V-film, while it is restricted only to the surface region for the bulk specimen.

### O9-4

#### INTERNAL FRICTION IN MEMBRANES AND THIN FILMS

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The internal friction of membranes is a new field in which the damping is controlled by the imaginary part of the internal stress. This situation contrasts sharply to traditional damping which is controlled by the imaginary parts of the elastic moduli. The damping in ultrathin composite films is controlled by both, the imaginary parts of the moduli as well as the internal stress. This is to say that thin film adhesion will also influence the dynamical properties of composite thin layer materials. The effect of these three features, the composite nature of thin films, the importance of internal stresses and the adhesion between layers, will be discussed in this presentation. All three cases will be illustrated by recent experimental results on electrodeposited CuNi multilayer membranes, NiTi shape memory thin films sputtered onto on  $\text{SiO}_2/\text{Si}$  substrates and MOCVD/MBE grown InP monolayers on GaAs.

O10-1  
EXCITATION AND PROPERTIES OF ZERO SOUND IN METALS

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The existence of the fast signals of the electron sound (ES) under the excitation of the flat surface of the ultrapure metal (Ga, W, Mo, Al) by the piezoelectric transducer has been revealed. These signals were observed in zero magnetic field at helium temperatures, including the superconducting state.

We have measured both the ES phase velocities  $V$  and the Fermi velocities spectrum and found  $V$  to be approximately equal to the maximum Fermi velocity along a given direction of propagation, and frequency independent (50 and 100 MHz) in a collisionless limit. In a weak magnetic field ( $\leq 3$  Oe)  $V$  increases by some percents with the increase of  $H$ . Also we have found the attenuation of ES to be practically frequency-independent. These results together with the theoretical treatment permit us to identify ES as zero sound (ZS).

ZS may be observed only in the multicomponent Fermi liquid (multiband anisotropic metal), where it represents the antiphase oscillations of the separate components densities. ZS oscillations are coupled with the elastic deformation and may be excited (received) via piezotransducers.

In the hydrodynamical limit ZS transforms into a "concentration" mode having another phase velocity and being the strongly damped electron analogue of the first sound.

The investigation of ZS amplitude versus temperature is a useful method of the electron relaxation study.

O10-2  
UNUSUAL ELASTIC BEHAVIOUR OF NORMAL STATE  $UPt_3$

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We report on extensive ultrasonic investigations of the elastic properties of the heavy fermion compound  $UPt_3$  in the normalconducting state. In particular, careful measurements of the temperature dependence of the ultrasonic attenuation and sound velocity were performed in the vicinity of 5 K, where long-range antiferromagnetic order of the very small magnetic moments has been claimed to occur. Anomalous hysteresis effects in ultrasonic properties were observed which point to a by far more complex relationship between lattice instabilities and the formation of the heavy fermion state than usually expected.

## LOW-TEMPERATURE ANOMALIES OF ELASTIC MODULI IN NIOBIUM OF DIFFERENT PURITY IN THE NORMAL AND SUPERCONDUCTING STATES

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Temperature dependences of elastic moduli in metals are determined at low temperatures by the lattice and electron components. According to the free electron model, the electron component increases with temperature as  $T^2$  and the lattice component at  $T \ll \Theta$  ( $\Theta$  - Debye temperature) decreases as  $-T^4$ . Superposition of these contributions would lead to appearance of maximum in temperature dependences of elastic moduli, but for the common metals the electron component is negligibly small and the maximum would be observed in the temperature range  $T \ll 1$  K. In case of the transition metals, the electronic component increases sharply (by decades) and the temperature dependences of elastic moduli are non-monotonic at the liquid helium temperatures. It is of particular interest to study the behaviour of elastic moduli in niobium where in the normal, mixed and superconducting states different densities of normal electrons are realized.

In this work at frequencies 45-350 kHz, the study of the temperature dependences of dynamic moduli in niobium crystals with  $R_{300}/R_{4,2} = 60; 290; 660; 970$  in the range 2.5-20 K is made. It is found that in the normal and mixed states the modulus temperature dependences show maximum. In the superconducting state the practically linear decrease of the moduli is observed when decreasing temperature below  $T_c$ . This result differs from that obtained earlier for high-purity niobium where in the same temperature region the non-monotonic dependence with minimum was found. The above results testify that the simple free electron model does not describe the electron contribution to the elastic moduli of the transition metals.

O11-1  
ELASTIC AND DISSIPATIVE PROPERTIES OF METALS WITH ULTRAFINE GRAIN  
STRUCTURE

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The elastic and dissipative properties of silver and copper polycrystalline materials with ultrafine grain structure (nano and submicrocrystals) are investigated in a wide temperature range by different acoustic techniques (inverted torsional pendulum, composite vibrator method, echo-pulse and high frequency resonance methods) at frequencies from 10 Hz to 10 MHz. It was found that as-prepared samples with ultrafine grain structure demonstrated very unusual behaviour of mechanical properties: sharp increase, in comparison with ordinary metals, of yield point and hardness and essential (up to 20%) decrease of effective elastic moduli. The dependence of these properties as a function of microstructure which could be changed by the heat treatment was studied. Measurements of the elastic and dissipative properties revealed an irreversible internal friction peak and accompanying increase of elastic modulus at the characteristic temperature corresponding to that of a structure rearrangement. The peak temperature and modulus change do not depend on a frequency, however depend on the chemical composition of the material. The anomalous elastic and dissipative properties observed are critically discussed in terms of the models describing elastic and anelastic phenomena caused by rearrangement in the grain boundary region. Special attention is paid to the role of internal stresses in such processes.

O11-2  
INTERNAL FRICTION ASSOCIATED WITH GRAIN BOUNDARIES IN Ni-Cr ALLOYS

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High temperature internal friction have been investigated in detail in Ni-20 at.%Cr alloys, both in single- and polycrystals, by using a low-frequency inverted torsional pendulum from room temperature up to 1273K. The internal friction spectrum of Ni-20 at.%Cr polycrystal is mainly characterized by two relaxation peaks upon heating (P1 peak ~950K) and cooling (P3 peak ~1100K) respectively, and a large hysteresis in the P3 peak temperature range between heating and cooling cycles. In some experimental conditions, another peak P2 was also observed upon heating in the temperature range higher than P1 peak. In the Ni-20 at.%Cr single crystal, only the P1 peak was observed during heating and cooling. However, when the single crystal was recrystallized at 1200K after cold-rolling, an internal friction spectrum identical to that of polycrystal was observed. Experimental evidences lead us to conclude that P3 peak and the large hysteresis are associated with the grain boundaries and the P1 peak is related to a relaxation process taking place in the grains.

Furthermore, metallographical observations in measured polycrystals and in the recrystallized single crystal revealed both the presence of discrete carbide precipitates at the grain boundaries. It was shown that the internal friction behaviour associated with the grain boundaries depends strongly on the microstructure of the grain boundaries. When the grain boundary is free of carbide precipitates, only the P3 peak was observed during heating and cooling. On the contrary, when the grain boundary contains continuous distribution of carbide precipitates, only the P1 peak was observed during heating and cooling. A continuous evolution of the high temperature internal friction, towards to a stabilized P3 peak, was also observed in an oxygen contaminated environment.

The obtained results lead us to attribute the P3 peak to grain boundary relaxation, which is affected by the carbide precipitates. The hysteresis can be due to changes in the grain boundary, for instance, carbide precipitation-dissolution.

### O11-3

#### THE STUDY OF INTERNAL FRICTION IN METAL-METAL LAYERED MATERIALS

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The behaviour of the interfaces is a fundamental parameter of microheterogeneous systems. The internal friction measuring technique is a very sensitive method to study the state of interfaces.

The aim of the present study was to investigate:

- the state of grain boundaries and their small distance sliding (grain boundary internal friction peak; GB peak),
- the dislocation motion by conservative or non-conservative way (low- and high temperature background; LTBG and HTBG),
- the existence of regularly spaced dislocation network (amplitude dependent internal friction).

The amplitude dependent and independent internal friction measurements were carried out in a torsion and in an inverted torsion pendulums over a temperature range of 300-700 K. The amplitude dependence was studied in the deformation range of  $10^{-5}$  -  $2.5 \cdot 10^{-4}$ . The metal-metal layered samples were produced by electrodeposition.

The main results on the areas mentioned above are as follows:

- 1) The atoms of the deposited layer diffusing into the substrate mainly along the substrate grain boundaries suppress the GB peak of the substrate. The effectiveness of this suppression is strongly dependent on the kind of the deposited metal.
- 2) The LTBG shifted parallelly to higher values of internal friction after the deposition. The rise of HTBG of metal-metal system is higher than the rise of the substrate HTBG.
- 3) Measuring the internal friction as a function of vibration amplitude we find a hysteresis on the curves detected on increasing-decreasing series of amplitude. This hysteresis disappears after the dissolving of the deposit layer. The area of the hysteresis depends on the kind of deposit metal and strongly decreases during the second series of amplitude-dependent measurements. The Granato-Lücke plot of this hysteresis shows that it can be resulted by a regularly spaced dislocation network.

### O11-4

#### ANELASTICITY OF ULTRA-FINE GRAINED POLYCRYSTALLINE GOLD

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Anelastic properties of ultra-fine grained gold produced by the gas deposition (GD) method, were investigated in the temperature range 78-820K. The GD method is a promising method to produce so-called nanocrystalline materials with grain size of about 10nm. In this method, ultra-fine particles produced in an evaporation chamber with a controlled He gas pressure are transported through a pipe to a deposition chamber where they are deposited on the substrate to form ultra-fine grained films. So far, the grain size of specimens observed by SEM was about 60nm. Internal friction ( $Q^{-1}$ ) and elastic modulus (E) were measured by means of flexural vibration (about 300Hz).

$Q^{-1}$  showed two small peaks at 130K and 210K and one pronounced peak at around 550K with large background increasing up to 820K. The 130K peak grew and was annealed out below 650K. The 210K peak was annealed out at around 400K. The peak temperatures of these two peaks agreed with neither the Bordoni peak nor the point defect-dislocation interaction peaks so far reported. The peak temperature of the 550K peak moved from 525K to 580K during thermal cyclings with increasingly higher temperatures and disappeared after annealing at 1123K. This peak could be identified as the ordinary low temperature grain boundary peak from the peak temperature of 580K and the recovery temperature observed in ordinary polycrystalline gold.

E showed a pronounced recovery (or increase) during annealings in the temperature range 600-700K. This was accompanied by a pronounced recovery (or decrease) in  $Q^{-1}$  around the grain boundary peak. It was rather surprising that the specimen remained brittle even after annealing at 1123K where recrystallization should be expected to occur from the disappearance of the low temperature grain boundary peak.

The present results on 60nm sized polycrystalline gold seem to be comparable in many respects to the results on 10nm sized nanocrystalline palladium by M. Weller et al. (1991).

**O11-5**  
**ANELASTIC RELAXATION STRUCTURAL STABILITY AND TRANSFORMATIONS IN  
NANOCRYSTALLINE METALS AND ALLOYS**

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Mechanical properties of nanocrystalline (nc) materials display increasing interest from a fundamental point of view as well as for their potential technological applications.

Two structural features appear of major concern in the phenomenological analyses of these properties: grains and boundary regions. The "grain size" strongly affects strength and deformation behaviour. Anomalous trends of some specific properties, e.g. strength, are observed when dimensionality effects affect the mechanism of defects generation and mobility. This is the case, in pure metals, when strength and plasticity depend on dislocation generation and dynamics. Moreover, since in nc materials an appreciable fraction of atoms are located in the regions within and near one or more boundaries, the "boundary regions" may be an important microstructural feature in controlling specific properties.

Mechanical spectroscopy techniques have been widely applied in the past years to study mechanical properties in polycrystalline materials. In particular grain boundary sliding mechanisms have been studied through measurement of the elastic energy dissipation. Moreover, detailed information on the elastic instability phenomena linked to structural transformations (grain growth, crystallization) were provided by dynamic elasticity moduli measurements.

In the present research elastic energy dissipation and dynamic Young's modulus measurements, coupled with TEM observations, have been performed on pure nc bulk metals, solid solutions and intermetallics, obtained by ball-milling plus cold consolidation. The anelasticity spectra of nc metals show noticeable differences with respect to those of polycrystals. In particular, strong reductions of the background damping level and a low temperature instability regime for the dynamic Young's modulus have been observed and correlated to the grain size and defect density. Anelastic relaxation effects have been observed just below the temperature where the elastic instability regime for the elasticity moduli sets up. Possible correlations of the relaxation with grain boundary or dislocation mechanisms and some similarity with the anelastic relaxation of nanostructured thin films deposited onto rigid substrate are emphasized.

**O11-6**  
**NONLINEAR MECHANICAL RELAXATION ASSOCIATED WITH THE VISCOUS SLIDING OF  
GRAIN BOUNDARIES**

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In 1947, Kê reported for the first time the results of his research on the mechanical behavior of grain boundaries in polycrystalline aluminium with anelastic measurements. Experimental results showed that the grain boundaries behave in a viscous manner and can slide under the action of shear stress. Because of the existence of grain edges and corners in a fine-grained polycrystalline specimen, the sliding cannot proceed unlimitedly. As a consequence, an internal friction peak (versus temperature) appears and exhibits a definite relaxation strength.

Recently, the internal friction of high-purity aluminium bicrystals with random boundary (mis-orientation angle  $60^\circ$ ) and with boundary having a coincident site lattice ( $\Sigma 11$ ) was measured with forced-vibration method. Internal friction peaks (versus temperature) were observed around  $200^\circ\text{C}$  with frequency of vibration 1 Hz. The activation energy concerned was found to be 0.88 eV. Since no grain edges and corners should exist in bicrystal specimens, it is considered that the basic process associated with the internal friction peak observed in bicrystals is the viscous sliding of the boundary dragging the dislocation substructure existed in the vicinity of the boundary. This dragging process controls the viscous sliding along the boundary so that an internal friction peak can appear. However, since the dislocation in the substructure can move during the boundary sliding, the limiting action is of a relaxation type instead of purely elastic as in the case offered by the grain edges and corners in polycrystalline specimens.

**O12-1**  
**THE DIAELASTIC EFFECT IN THERMODYNAMIC PROPERTIES OF CONDENSED MATTER\***

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Relaxation and Resonance Effects are well known in the Internal Friction and Ultrasonic Attenuation literature. In recent years a new kind of response, the Diaelastic effect, has become useful in the characterization of defects in solids. The diaelastic effect is analogous to the diamagnetic effect for magnetic materials, and corresponds to the stress induced generation of dipoles in the crystal, in contrast to the stress-induced ordering of preexisting dipoles, which leads to relaxation effects. The response is temperature and frequency independent, but does depend on polarization. The latter feature makes it useful in characterizing the symmetry of defects. Examples for different crystal types are discussed. The known diaelastic response of crystals containing interstitials has been used to construct a model according to which liquid and amorphous metals are crystals containing a few percent of self-interstitials. The phenomenological aspects of the model do not depend, however, on the microscopic configuration, but only on the diaelastic properties known experimentally to be universal for amorphous materials.

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**O12-2**  
**INFLUENCE OF A DISTRIBUTION OF RELAXATION TIMES IN THE DAMPING OF MATERIALS**

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The dynamic response of a linear viscoelastic solid is clearly affected by the presence of a distribution function of relaxation times, even in the case of low relaxation strengths. The functional forms of the distribution functions and their characteristic parameters are related to the relaxation process, and the determination of these parameters gives valuable information about the mechanisms involved in the material.

The *tangent distribution function*, which was introduced in a recent work, allows to include the internal friction of a linear viscoelastic solid in the group of magnitudes which can be expressed by distribution functions, as the modulus or the compliance, either in terms of relaxation times or in the complementary variable, the excitation frequency. This leads to different procedures for analyzing the internal friction behaviour, both as a function of temperature or frequency. The concepts developed in this work are applied to experimental data for the Snoek relaxation in Nb-O alloys.

O12-3

ACOUSTIC EMISSION AND SELF-ORGANIZED CRITICALITY ASSOCIATED TO  
FRACTURE PROCESSES DURING HYDROGEN PRECIPITATION IN Nb

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Acoustic emission activity has been detected in niobium during hydrogen precipitation from solid solution. The observed emission is attributed to the nucleation and propagation of microcracks generated by the misfitting growing precipitate particles. The analysis of the acoustic signals reveals that the distribution of their amplitudes follows the Gutenberg-Richter law of earthquakes  $N(A > A_0) \propto A_0^{-c}$ ,  $c = 0.9$  over more than two orders of magnitude. The observed power law cannot be explained in terms of a critical phenomenon because hydride precipitation is a first order transition not implying fluctuations over all scales. Rather, it indicates that during the fracture processes caused by the precipitation, the Nb-H system goes into a self-organized critical state similarly to what occurs in the earth crust as far as the earthquakes are concerned.

### O13-1

#### MAGNETOPLASTIC EFFECT IN NON-MAGNETIC CRYSTALS AND INTERNAL FRICTION

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Displacements of dislocations as a result of exposing the specimens to a static magnetic field ( $B=0.1-2T$ ) is found and investigated for NaCl, CsI, LiF, Zn and Al single crystals. This effect is characterized by the following properties: 1) the mean dislocation path linearly increases with the time of the magnetic treatment; 2) the mean dislocation velocity is proportional to the square of the magnetic induction and inversely proportional to the square root of the concentration of paramagnetic impurities; 3) the effect is temperature independent in the range  $T=4.2-77K$  and it grows only by 20-30% with a further increase of  $T$  up to 293K; 4) an abrupt decrease of the effect is found when the frequency  $\nu$  of an alternating magnetic field exceeds some critical value  $\nu_0 \propto B^2$  ( $\nu_0 \sim 15Hz$  at  $B=0.3T$ ); 5) a strong effect of a static electric field ( $E \sim 2-20V/cm$ ) on dislocation mobility in NaCl specimens simultaneously exposed to a static magnetic field is found (with no electric influence at  $B=0$ ). The phenomenon is interpreted as a result of unlocking of dislocations from paramagnetic centers under the action of magnetic field and motion of dislocations in the internal stress field. Possible manifestations of the magnetoplastic effect in the internal friction phenomena are discussed.

### O13-2

#### GIANT MAGNETOSTRICTIVE MATERIALS: THIN FILM FORMATION AND APPLICATION TO MAGNETIC SURFACE ACOUSTIC WAVE DEVICES

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The giant-magnetostrictive  $DyFe_2$  and  $TbFe_2$  intermetallics were pulverized by cyclic hydriding and dehydriding reactions. The produced powders were flashly evaporated to form thin film  $Tb_{0.3}Dy_{0.7}Fe_2$ . The film formation was examined in different vacua, deposition rates and substrates temperatures. The  $Tb_{0.3}Dy_{0.7}Fe_2$  film exhibited quite different M-H curves and magnetostrictions depending on substrate temperature and vacuum to deposition rate. This paper demonstrates the marked effect of film structure and composition on the magnetostriction. The results obtained are important to assess the magneto-elastic interactions of magnetostrictive  $Tb_{0.3}Dy_{0.7}Fe_2$  thin films for magnetic surface acoustic wave(MSAW) devices.

O13-3  
EFFECT OF HIGH-FREQUENCY PHONONS ON THE MAGNETIC MOMENT OF  $\text{FeBO}_3$

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By means of a SQUID magnetometer we have investigated the change of the magnetic moment  $\Delta M$  due to the excitation of high-frequency (0.5-2.5 GHz) phonons in the easy-plane antiferromagnet  $\text{FeBO}_3$ . These phonons were excited at helium temperature both by conventional magnetic resonance and parallel pumping techniques [1] via magnetoelastic interactions. In the low-field range ( $H < 150$  Oe) excitation of phonons results in a decrease while in the high-field range ( $H \geq 150$  Oe) excitation leads to an increase of the magnetic moment. The observed effects can be qualitatively described by the excitation of standing magnetoelastic modes which, in principle, would decrease the magnetic moment of the sample. The most important relaxation process of these modes is based on three-particle interactions with the magnon branch [2]. A theoretical analysis of the contribution to  $\Delta M$  arising from secondary quasi-particles shows that the excitation of non-equilibrium magnetoelastic modes leads to an increase of the total magnetic moment. The competition of these two contributions can explain the observed field dependence of  $\Delta M$ . While in conventional resonance experiments the observed change of  $\Delta M$  showed a linear dependence on the absorbed power, in parallel pumping experiments a strongly nonlinear behaviour was found close to the threshold. A detailed analysis shows that the threshold is essentially determined by the relaxation of a directly excited nonthermal packet of magnetoelastic waves.

We discuss the possibility of applying this effect for detecting high-frequency phonons in technical applications. The limiting frequency of such a small-bandwidth detector was calculated to be about 4 GHz. The sensitivity was evaluated from experiment to amount to  $5 \cdot 10^{-6}$  Oe  $\text{cm}^3$  for an elastic excitation power of 0.1 mW.

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O13-4  
THE ULTRASONIC BEHAVIOUR OF FERRIMAGNETIC GARNET YIG CRYSTAL UNDER PRESSURE

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ABSTRACT

Similar ultrasonic behaviours whether under atmosphere or under pressure have been found from the measurement on sound velocity and attenuation of garnet YAG and YIG. Obviously it was attributed to same garnet crystal structure. But, the ultrasonic behaviours of ferrimagnetic garnet YIG and nonmagnetic garnet YAG were different as external magnetic field was applied. This paper introduces ultrasonic behaviour of ferrimagnetic garnet YIG crystal under pressure, which reveals the fact that there exists magnon-phonon interaction in YIG.

We found that whether in the transverse wave or longitudinal wave and whether for sound velocity or attenuation too, an anomaly phenomenon appeared with pressure as external magnetic field was applied. The attenuation was exhibited the pronounced peak with pressure and the sound velocity was exhibited obvious plateau with pressure. This is different from to that without external magnetic field.

## O14-1 RECENT APPLICATIONS OF HIGH-DAMPING HYSTERETIC DEVICES FOR THE SEISMIC ISOLATION OF BUILDINGS, BRIDGES AND INDUSTRIAL EQUIPMENT

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Seismic isolation is a technique in which a structure is decoupled from earthquake-induced ground motions. In New Zealand, Italy, the USA and Japan this technique has now advanced to the point where it is often considered for the protection of both new and existing buildings, bridges, and to a lesser extent, industrial plant.

Seismic isolation systems have two important functions:

- The period of the isolated structure is increased to a value beyond that which dominates in a typical earthquake
- The displacement is controlled (to 50-200 mm) by the addition of an appropriate amount of damping (often approximately 20 percent of critical).

The increased period (>1.5 seconds) is achieved via a flexible support which provides a reduction in the 'stiffness' or 'spring constant' between the structure and the ground. Examples are flexible piles and rubber elastomeric bearings. The damping is usually predominantly hysteretic, provided by plastic deformation of either steel, lead or rubber at strain amplitudes of up to 100 percent.

Our studies of seismic isolation began in 1968 as the synthesis of two groups working on materials science and engineering seismology respectively. Our research has had three main components: experiments, theoretical work and application of seismic isolation devices. Devices invented and developed in our laboratory, and successfully applied in real seismic isolation systems, include various designs of steel damper, the lead-extrusion damper and the lead-rubber bearing. A paper describing this work was delivered at the 8th ICIFUAS conference in 1985 and we have recently (1993) produced a book "An Introduction to Seismic Isolation" describing the technique and devices in detail.

In this paper we present a critical analysis of the various damping devices and a summary of their application worldwide to date.

## O14-2 SCANNING ACOUSTIC IMAGING OF STRESSES

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A new acoustic technique for mapping stresses in isotropic, solid materials is based on a comparison of the amplitudes of polarized modes propagating through the stress induced anisotropic areas with those propagating through stress-free volumes of the sample. Acoustic images display variations in the stress field utilizing differences in acoustic wave amplitude often enhanced by interference effects. The presence of internal stresses in the interior of isotropic, solid materials may be understood, in analogy to optics, as a temporary or artificial birefringence. This effect normally persists while the loads are maintained but vanishes when the stress is removed. This phenomenon is known as a temporary or artificial double refraction and was first observed by D. Brewster in 1816 in optically transparent materials. The corresponding effect in acoustics is known as acoustic birefringence or trirefringence in isotropic materials subjected to stress.

In acoustic microscopy, every shear mode created by mode conversion of a longitudinal refracted wave at the water-solid interface is polarized. Following the analogy to optics, in the stressed areas, the polarized shear mode will experience birefringence. The shear wave will split into two, orthogonally polarized components propagating with different speeds inside the stressed area. The waves received from the stressed area will show a decrease in intensity compared to that of the arrivals from the isotropic, stress free volumes. This happens, because the water-solid interface in an acoustic microscope can be compared to an optical analyzer, which allows only certain polarizations to return to the receiver through the layer of water. In contrast to light waves, acoustic waves show trirefringence, because the speed of the longitudinal wave is also affected by the acting stress. The broad range of wave-lengths available in acoustic imaging makes this technique feasible for many practical applications.

A few examples are given: a disc under diametral concentrated load, polymer and aluminum samples under compression, and metal and ceramic samples with residual stresses illustrate the advantages and limitations of this technique. In case of the diametrically compressed disc, the analytically obtained solution for the sums and the differences of the principal stresses for the sample used in the experiment are compared with the acoustic images.

#### O14-3

### DETERMINATION OF ELASTIC CONSTANTS OF SINGLE CRYSTALS BY THE RECTANGULAR PARALLELEPIPED RESONANCE METHOD

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The rectangular parallelepiped resonance method is a useful method for determining elastic constants of single crystals. The great advantage of this method is that one can use a very small specimen of about 2 mm in edge length. However, the numerical analysis to obtain the values of the elastic constants from the observed resonance frequencies is very laborious. A new method of analysis is proposed, which is applicable to specimens of cubic crystals. With this method, we have determined the elastic constants  $C_{11}$ ,  $C_{12}$  and  $C_{44}$  of a series of Ni-based  $L_{12}$  compounds  $Ni_3X$  with  $X = Mn, Fe, Al, Ga, Ge$  and  $Si$  from room temperature to 1000°C.

The number of independent elastic constants is larger for crystals with lower symmetry: 3 for cubic, 5 for hexagonal, 6 or 7 for tetragonal and 9 for orthorhombic crystals. Since the analysis is made essentially by trial and error, the time required to reach the final solution becomes longer for crystals with low symmetry. Attempts are being made to determine directly the vibrational mode of each peak in a resonance spectrum. Such information is expected to help greatly the process of analysis for evaluating the elastic constants.

#### O14-4

### MEASUREMENTS OF INTERNAL FRICTION IN POLYCRYSTALLINE MATERIALS USING LASER ULTRASOUND

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In polycrystalline materials, the internal friction depends on dislocation damping, Bloch wall movements, thermoelasticity and other mechanisms. Hence, absorption measurements should be helpful in order to detect, for example, changes in the dislocation structure taking place during plastic deformation by (thermo)mechanical treatment, creep or fatigue. However, precise measurements of ultrasonic absorption or internal friction in the frequency range from 0.1 - 10 MHz in polycrystalline materials like steels, are usually not possible by means of conventional ultrasonic techniques, because the scattering of ultrasound caused by the grain boundaries is much larger than the inelastic part, i.e. the internal friction.

Precise measurements of ultrasonic absorption are possible by measuring the reverberation of ultrasound in specimens of limited size. If piezoelectric transducers are used for generation and detection, the effect of mechanical damping by the transducer has to be taken into account, especially for specimen volumes below 10 cm<sup>3</sup>. We have extended the reverberation technique allowing non-contact measurement of the ultrasonic absorption by using laser ultrasound, thus avoiding any external damping of the reverberation signal. In addition, the new technique enables one to carry out absorption measurements up to very high temperatures, and it can be applied to samples of complex shape. Laser ultrasonics also allows one to measure the elastic moduli as a function of temperature. In this paper, the measurement technique is described and examples for internal friction measurements together with the elastic moduli are presented for polycrystalline steels, copper, aluminum, and ceramics as a function of deformation, frequency, and temperature. In those polycrystalline metals where internal friction is caused to a large extent by movement of dislocations, the internal friction does not obey the usual frequency dependence expected from a Debye behavior, i.e.  $Q^{-1} \sim \omega$  but  $Q^{-1}$  is rather constant. However, the contribution from the magnetoelastic effect in ferritic polycrystalline steel does behave like a Debye relaxator. Furthermore, in white cast iron, a ferromagnetic phase transition at 480 K is observed which is related to the Curie point of the cementite phase. Possible origins of the various contributions to  $Q^{-1}$  will be discussed.

O14-5  
RESONANT ULTRASOUND SPECTROSCOPIC TECHNIQUES FOR MEASUREMENT OF THE  
ELASTIC MODULI OF SOLIDS

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The mechanical resonant response of a solid depends on its shape, density, elastic moduli and dissipation. We describe the instrumentation and computational methods for acquiring and analyzing the resonant ultrasound spectrum of very small ( $0.001 \text{ cm}^3$ ) samples as a function of temperature, and provide examples of measurements on ceramics, metals and superconductors to illustrate the range of information that can be obtained. The information acquired is in some cases comparable to that obtained from other more conventional ultrasonic measurement techniques, but resonant ultrasound spectroscopy (RUS) is unique in that all moduli are determined simultaneously to very high accuracy without any transducer bond corrections. Thus in circumstances where high relative or absolute accuracy is required for very small crystalline or other anisotropic samples RUS can provide unique information.

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O14-6  
INTERNAL STRESS EVALUATION FROM MECHANICAL BARKHAUSEN EFFECT

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New method - based on the analysis of the Mechanical Barkhausen Effect (MBE) noise measurements was used to reveal the distribution of internal stresses in ferromagnetic materials in some Fe-Ti ferrous alloys. Intensity of MBE noise has been measured for wire samples as a function of the applied shear stress using inverted pendulum. Measurements of MBE were followed by measurements of the magnetomechanical damping. It was revealed that the integral of hysteresis loop  $U(\gamma)$  of MBE noise intensity (after correction on strain rate) is simply proportional to the energy losses for one cycle, calculated from internal friction. The dependence of the integral of the MBE hysteresis loop  $U(\gamma)$  on the shear strain amplitude  $\gamma_0$  - function  $U(\gamma_0)$  - was then used to find experimental curve of the internal stress distribution function  $N_\epsilon(\sigma)$ . These integrals as calculated for shear vibration have been corrected to the uniform stress mode of vibration. It was tested that  $N_\epsilon(\sigma)$  function can be fitted by some phenomenological function  $N_p(\sigma)$ , which was proposed previously by the Birchak and Smith's model of magnetomechanical hysteresis process.

#### O14-7

### N-AGEING IN ALUMINISED STEEL SHEET: AN INDUSTRIAL APPLICATION OF HIGH FREQUENCY INTERNAL FRICTION MEASUREMENTS TO POINT DEFECT/INTERFACE INTERACTIONS

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Aluminised sheet (two-sided, with a 80-150g/m<sup>2</sup> Al-9%Si alloy coating), obtained by a continuous hot-dipping process of steel sheet, is widely used in the automotive and building industries because of its favorable high temperature corrosion resistance combined with a high reflectivity and a good formability. It has been known for some time that cold rolled Al-clad sheet, containing a controlled amount of interstitial nitrogen before cladding, is resistant to the formation of brittle Fe-Al intermetallics, which give the material a poor high temperature performance. The aim of the present research was to apply a similar method to the improvement of the high temperature resistance of continuously aluminised sheet and to determine the precise nature of the mechanism responsible for the better performance.

The N-ageing of aluminised sheet was therefore studied by means of the APUCOT, a high frequency internal friction measuring device based on the four component resonant bar technique resonating longitudinally at 40kHz.

Aluminised sheets with substrate compositions giving a large difference in high temperature performance, were heat treated and the kinetics of the N-ageing were studied.

Microanalytical studies using AES showed that the coating/substrate interface acts as an effective trap for N-interstitials.

In view of the stability of AlN, the results strongly suggest that a thin AlN-diffusion barrier is formed at the coating/substrate interface when the kinetics of the Fe-Al intermetallics formation are slower than the kinetics of the diffusion barrier formation. The latter is essentially N-diffusion controlled.

Based on the results it is possible to define the aluminised sheet substrate chemistry that will yield a coated sheet with a high temperature resistance up to the melting point of the alloy coating.

#### O14-8

### ANALYSIS OF THE ANELASTIC CREEP OF Al AND TWO Al-Cu ALLOYS

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The future nanometer-dimension technology requires highly stable and drift-free materials. Therefore, anelastic creep of pure Al, AlCu and AlCuMg has been measured by means of a high resolution laser heterodyne interferometer. The anelastic deformation has been studied as a function of time for several thermomechanical treatments. The obtained creep functions closely follow over several time decades a power law with exponents between 0.3 and 0.5. The total deformation of pure Al also contains an irreversible contribution following a logarithmic creep law. The relaxation strengths strongly depend on the material and the thermomechanical treatment. The microstructures resulting from various treatments and observed in TEM can be associated with the different anelastic behaviours. Recognizing the characteristic times involved in the deformation-time curves was not univocally possible, due to the difficulties of a spectral decomposition both in the discrete and in the continuum. As the operation of inverting a Laplace transform is substantially unstable, the form of the spectrum is shown to depend strongly on the features of the functions used to fit the experimental data. Moreover, the different phenomena responsible for anelastic creep could not directly be identified on the deformation-time curves which do not present any inflexion or reference point. Nevertheless, it has been possible to quantitatively compare the static anelastic behaviour with the dynamic response of the same materials, measured as a function of the temperature or frequency at different temperatures. A good agreement between the two types of results has been obtained. With respect to the interpretation of the results, static and dynamic methods appear to be complementary. Direct creep measurement provides information on long relaxation times phenomena and allows to distinguish between reversible and irreversible contributions, whereas dynamic methods allow a better separation of the dissipation mechanisms. In particular, for artificially aged AlCu, the static behaviour of the alloy could be predicted from the internal friction measured as a function of temperature. The nearly perfect correspondence between the model and the experimental creep data allows its static behaviour to be interpreted in terms of a Zener mechanism and a  $\theta'$ -peak. Microstructural TEM observations confirm this interpretation.

**PI-1**  
**INTERACTION OF DISSOLVED ATOMS AND HYDROGEN SNOEK RELAXATION IN Nb AND Ta (COMPUTER SIMULATION)**

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Internal friction due to "diffusion under stress" of H and D atoms was calculated in Ta(Nb)-O(N)-H(D) alloys to use internal friction for verification of the applicability of the model of interaction of dissolved interstitial atoms and also to investigate the relaxation mechanism. A long-range strain-induced (elastic) interaction model supplemented by repulsion interaction in the nearest coordination shells was used. The short-range order in interstitial solid solution was simulated by Monte-Carlo method. It was supposed that the short-range order effects internal friction by changing the energy of H or D atoms in solid solution and therefore the activation energy of the relaxation process.

Computer simulation was carried out with concrete values of energies of elastic interaction and with different radii of additional repulsion interaction. The calculated spectra are in a good agreement with experimental data when the repulsion interaction expands up to the third coordination shell. It was shown that the used strain-induced interaction model supplemented by the repulsion in three nearest shells are useful for description of the solid solutions and the relaxation mechanism consists in reorientation of H(D) atoms in the third coordination shell near O(N) atoms.

**PI-2**  
**HYDROGEN AND OXYGEN-RELATED ANELASTIC EFFECTS IN THE Ta<sub>75</sub>Nb<sub>25</sub> ALLOY**  
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The Gorsky relaxation due to long range diffusion of H has been investigated over a wide range of temperatures by elastic after-effect. Contrary to what happens in pure Ta and Nb, in the Ta<sub>75</sub>Nb<sub>25</sub> alloy the hydrogen diffusion coefficient displays an Arrhenius type of temperature dependence between 125 and 415 K. The hydrogen precipitation relaxation has also been observed, together with a lower temperature anelastic effect. It is unclear whether the lower temperature effect is a Snoek or an O - H pair reorientation relaxation.

The oxygen Snoek effect is associated with a wide spectrum of relaxation times, the origin of which is related to the multiplicity of interstitial sites available to O in the alloy.

### P1-3

## TEMPERATURE DEPENDENCE OF THE INTERNAL FRICTION AND MODULUS CHANGE IN $Y_2O_3$ OR $MgO$ DOPED $ZrO_2$ POLYCRYSTALS

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Internal friction and frequency measurements were carried out in  $Y_2O_3$  or  $MgO$  doped zirconia polycrystals at low frequency (1,5 - 3,5 Hz), in the temperature interval from 130 to 623K. The results obtained with the first system confirm those of the literature. In the  $ZrO_2$ - $MgO$  system, two peaks have been observed, at 223 and 455K, respectively. The first is sensitive to linear annealings at temperatures between 473 and 573K, and decreases with  $MgO$  concentration, whereas the second appears for higher concentrations and increases with them. This last peak has been ascribed to the reorientation of oxygen vacancy-magnesium cation pair in the cubic phase, induced by stress, with an activation energy of  $\sim 1,17$  eV.

### P1-4

## ANELASTIC RELAXATIONS DUE TO INTERSTITIAL IMPURITIES IN NIOBIUM AND TANTALUM

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Measurements of internal friction constitute an important tool for study of anelastic relaxations when interstitial impurities are present in a metallic matrix. One of these processes observed by such measurements is known as stress induced ordering. This work shows the study carried out in polycrystalline samples of niobium and single crystals samples of niobium and tantalum, pure and doped with heavy interstitials as oxygen and nitrogen. The experimental data are obtained by internal friction measurements as a function of the temperature. The results show relaxations structures, which are decomposed in their constituted Debye peaks. These peaks are related to relaxations processes to stress induced ordering of interstitial atoms around the metallic matrix. (Financial support: CNPq and FUNDUNESP).

#### PI-5

### INTERNAL FRICTION IN PURE IRON NITROGENATED BY DIFFERENT METHODS

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A method for the determination of Nitrogen in solid solution using internal friction in ion implanted pure Fe, is presented. It allows also the indirect determination of some combined nitrogen (Fe nitride concentration).

It consists on measuring the Snoek relaxation during isothermals at low frequencies in a sub resonant forced pendulum. The polycrystalline samples are nitrogenated by ion implantation or by annealing in ammonia atmosphere.

Chemical analysis by vacuum fusion technic are necessary to check the proportionally factor between the internal friction height and the interstitial nitrogen content. It is assumed that the samples are not textured and that the nitrogen is completely dissolve by an annealing in inert atmosphere.

This kind of evaluations could be useful for characterizing nitrogenated bcc metals in relation to certain applications where mechanical properties could be altered by the free nitrogen content.

#### PI-6

### RELAXATION SPECTRUM AND PROPERTIES OF MARAGING STEELS WITH SILICON

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The temperature dependence of internal friction (IF) of Fe-Cr-Ni alloys with different silicon content have been investigated. The measurements of IF were carried out for plasma powder spraying specimens. A resonance kHz-installation (amplitude deformation  $10^{-6}$ - $10^{-7}$ , temperature range 20 - 700°C) and low-frequency reverse torsion pendulum were used for investigation.

The temperature spectrum of IF was studied for specimens, first, after spraying and second, after heating at 100 - 800°C temperature range for 1,5 hour. The relaxation maximum of internal friction at 50°C ( $H \sim 13$ -14 kcal/mol) and phase peak at 550°C were revealed at 1 kHz. The shape and parameters of maximum depend on heat treatment regimes. The peak nature of IF in connection with mechanism of solid solution decomposition of the spraying materials during heating was discussed.

The hardening of Fe-Cr-Ni-Si alloys during heating has been achieved because of the precipitation of intermetallic compound phases of  $Ni_3Ti$  type.

**P1-7**  
**Q<sup>-1</sup> SPECTRA CONNECTED WITH C UNDER SOLUTE ATOM INTERACTIONS**

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Dynamic modulus and Internal friction spectra vs temperature, have been examined in 11% Cr steel (MANET) after quenching with different cooling profiles from 1348K, in the field of austenite, and above, at 1473K.

The results are interpreted by referring the various Q<sup>-1</sup> peaks, of the Snoek type, to the different numbers n, from 0 to 6, of the Cr atoms which may occupy the corners of interstice with C, i.e. to C-nCr associates with geometry tending to octahedral in martensite.

Neglecting diffusive motion of the solute atoms during quenching the associates in martensite are considered directly bounded with corresponding associates formed in the fcc structure of austenite.

A statistical model is discussed according to which C-6Cr associates form or disappear below or above a critical temperature T<sub>c</sub> in the γ-phase: for a preferential bonding energy of the Cr atoms to C consistent with the activation energies characterizing the Q<sup>-1</sup> peaks, it results T<sub>c</sub> to be in the austenite range. The model is illustrated by snapshots obtained with the Monte Carlo method.

Q<sup>-1</sup> spectra obtained after quenching from T > T<sub>c</sub> or T < T<sub>c</sub> are presented, which confirm this interpretation. Further observations regard effects of thermal treatments at high temperatures, with slow cooling down to the γ-phase limit and subsequent quenching to room T. The results are consistent with the formation of C-6Cr associates with characteristics of the bcc structure, which may be taken as indication of a high T stage of martensite nucleation.

The Internal friction and dynamic modulus measurements have been accompanied by SEM metallography, mechanical tests and analyses of the X-ray diffraction lines, in general consistent with the above mentioned interpretations.

**P1-8**  
**STABLE RELAXATIONS IN MULTIPHASE Al-Zn ALLOY**

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The experiment on a forced vibration pendulum reveals the stable damping in a multiphase Al-Zn eutectoid alloy as three kinds of relaxation processes: One is linear, and two are nonlinear. The characteristics of frequency, amplitude, temperature, and process activation energy for each kind of relaxation are presented and the correlation among those relaxations are discussed as well.

**P1-9**  
**ANALYSIS OF THE SNOEK RELAXATION IN BCC METALS**

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It is normally assumed in the literature that the Snoek relaxation in b.c.c. metals leads to an internal friction peak of the Debye type, at very low interstitial contents. By means of a new measuring technique, however, it is shown that this is not generally the case. This technique allows a simultaneous measurement of the internal friction against the temperature and of its derivatives with respect to the temperature and the frequency. The data indicates that a distribution of relaxation times is present, even for concentrations of few hundreds atomic ppm.

Some results for the Snoek relaxation in niobium are discussed in detail, showing that the parameters of the distribution change with the concentration of interstitials. Finally, the physical parameters that characterize the internal friction peak are discussed.

**P1-10**  
**COMPUTER SIMULATION OF THE STRENGTH OF SNOEK RELAXATION IN TERNARY BCC ALLOYS**

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Atomistic simulation of interstitial (i) and substitutional (s) impurity atom pairs in bcc Fe has been performed to estimate the relaxation strength associated with such pairs. A model bcc crystal containing an i atom and an s atom has been constructed by adopting empirical pairwise interatomic potentials, and stable configurations have been obtained by molecular statics. The energy and displacement field have been calculated for various neighbour configurations of the i-s pair. When the potential functions are chosen so as the equilibrium distance between the i and s atoms to be shorter than that between the i and the host atoms, the anisotropy of the strain field and so the relaxation strength is smaller than for an isolated i atom. The Snoek relaxation profile can be calculated theoretically from the strain field parameters and the binding energy obtained from the simulation. By comparing the relaxation profiles observed for dilute Fe-Mn-C alloys with calculated ones, the Mn-C binding energy has been estimated to be of the order of 0.1 eV.

**P1-11**  
**ZENER RELAXATION IN Cu-Al SINGLE CRYSTALS**

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The Zener peak was studied in Cu-Al  $\langle 111 \rangle$  single crystals with Al contents ranging between 3 % at. and 19 % at., by isothermal internal friction measurements in a large frequency range ( $10^{-4}$  - 160 Hz).

Between 500 K and 700 K, the relaxation strength has been observed to increase with temperature, not obeying the classical Curie-Weiss law generally observed in solid solutions.

The activation parameters : limit relaxation time and activation enthalpy have been accurately measured and found to vary with the aluminium content.

The influence of the aluminium content on the relaxation strength was observed and it was shown that the widely used LeClaire-Lomer relationship is not obeyed except in the lower temperature range.

**P1-12**  
**ANALYSIS OF SQUARE FREQUENCY SPECTRA AS A TEMPERATURE FUNCTION**

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In almost every work showing internal friction as a function of temperature, their authors present only the results for  $f^2 \times T$  with no analysis or comment. In this work, we have proposed a relation between frequency square and temperature for anelastic relaxation in solids. We have also verified experimentally our theory for niobium and tantalum doped with oxygen and nitrogen at low concentrations. (Financial support: CNPq and FUNDUNESP).

# P1-13

## INTERNAL FRICTION COMPREHENSIVE PROBING OF DEFECTS IN III-V SEMICONDUCTORS

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Internal friction (IF) measurements in the frequency-temperature domain bounded by 5 Hz, 50 kHz, 77 K, and 600 K are shown to give rich information about defect structure of III-V semiconductors.

Precipitates of the metal component of a III-V compound manifest themselves in characteristic IF  $\lambda$ -peaks arising in frequency range of a few Hz at the melting point of the metal. Concentration of the precipitates can be evaluated from height and form of the peak.

Point defects created due to ion implantation of B, P, As, Zn, and Fe lead to appearing of corresponding IF maxima.

Existence and state of a cracked surface layer (created e.g. by mechanical polishing) are also reflected in specific IF peculiarities arising probably due to a non-conservative motion of tips of microcracks in this layer.

Defects or impurities with deep electronic levels, which compensate a semiconductor making it to be high-resistive, are accompanied by fine Debye-form maxima of IF in kHz area. Characteristics of the maxima are straightly connected with electron structure of the defects and electric properties of a sample. The physical origin of this phenomenon is relaxation of a piezoelectric field (and, in turn, of an elasticity modulus - through the reverse piezoelectric effect) involving thermal emission of charge carriers from the deep levels to a band.

# P1-14

## MECHANICAL LOSS MEASUREMENT ON YTTRIA AND CALCIA STABILIZED ZIRCONIA

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Atomic defects in cubic  $ZrO_2$  are a direct consequence of doping with lower valent cations (10 mol %  $Y_2O_3$  or 16 mol %  $CaO$ ) which are required for stabilization of the cubic phase. Mechanical loss measurements were applied on single crystals of cubic zirconia to study the local crystallographic structure of atomic defects. Specimens with different longitudinal axes ([100], [110], [111]) were measured in torsional ( $f \approx 1$  Hz) and flexural oscillations ( $f \approx 1$  kHz).

A composite loss maximum consisting of two submaxima is observed: I ( $\approx 410$  K) and  $I_A$  ( $\approx 510$  K) in  $ZrO_2-Y_2O_3$ ;  $I'$  ( $\approx 430$  K) and  $I_A'$  ( $\approx 515$  K) in  $ZrO_2-CaO$  ( $f \approx 1$  Hz). The peak heights of maxima I and  $I'$  depend on specimen orientation as expected for a defect of trigonal ([111]) symmetry. This strongly points to oxygen vacancies located at nearest neighbour sites at the dopant atoms which form elastic (and electric) dipoles, i.e.  $(V_O \cdot Y_{Zr})$  or  $(V_O \cdot Ca)$ -pairs, which are aligned parallel to  $\langle 111 \rangle$  directions. The shape factor of the strain ellipsoid of the dipoles characterizing the anisotropy and strength of the local atomic displacements was determined as  $\delta\lambda \approx 0.12 - 0.14$  for yttria stabilized and  $\delta\lambda \approx 0.05$  for calcia stabilized zirconia. Loss maxima  $I_A$  and  $I_A'$  are assigned to larger clusters of oxygen vacancies with two or more dopant atoms.

PI-15  
INTERNAL FRICTION AND "S-I" INTERACTION IN NIOBIUM-BASED ALLOYS

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Known literature data about substitutional and interstitial "s-i" atom interaction were received by modelling method, as well as by comparing experimental results of oxygen atom thermodynamic and diffusion behavior in solid solution of VA group metal alloys with theoretical models based on trap concepts /1/. Thus received the "s-i" solute binding energies  $\Delta E_x$  for V-O pairs in Ta are not in good agreement quantitatively, and for Mo-O in Ta differ qualitatively.

In the present work the internal friction method /Snoek relaxation/ was used for investigation of Mo-O and V-O interaction in Nb. The substitutional solutes concentration  $C_s$  was varied from 0 to 50 at.%. The complex logarithmic decrement  $\delta$  curves were analysed by a computer and individual Gauss form peaks were obtained as a result. The last were accounted for by oxygen atoms in octahedral sites, having 1-nearest substitutional neighbours ( $l=0,1\dots6$ ).  $\Delta E_x$  were calculated by using received data about oxygen atom concentration in 1-position ( $C_1$ ) and the latter concentration  $V_1$  in alloys with different  $C_s$ .

Nb-Mo-O system. Mo-O interaction - weak repulsion,  $\Delta E_{\text{Mo-O}} = +0\dots2\text{kJ/ml}$ . The increase of  $H_{\text{dif}}$  of O-atoms with Mo being added to Nb is due to change of the saddle point energy ( $\Delta E^s$ ), but not to  $\Delta E_x$ . Numerical data  $\Delta E_x$  for different complexes were obtained.  $H_{\text{dif}}$  coincide with  $H_{\text{max}}$  for corresponding peaks.

Nb-V-O system. V-O interaction - strong attraction, chemical by nature and of local action.  $\Delta E_{\text{V-O}} = -46\text{kJ/ml}$  and  $\Delta E_{2\text{V-O}} = -61\text{kJ/ml}$ . The attraction inside complexes at  $l \geq 3$  is markedly weakened, local action is lost and O-atoms distribution approaches the equilibrium one. The oxygen jumps around the traps are taking place with the decrease of potential barrier on  $\Delta E''$ , but when escaping from trap the barrier increases on  $\Delta E'$ . The values of  $\Delta E'$  and  $\Delta E''$  were derived. As to the structure of potential barrier  $H_{\text{dif}} \neq H_{\text{max}}$ .

The obtained data showed different concentration changes of spectra  $\delta$  in alloys with weak and strong interactions, explained peculiarities of their investigation with different methods, discrepancies of literature data, the reasons of most model imperfections, etc.

RELAXATION STRENGTH OF POINT DEFECTS AT ARBITRARILY HIGH CONCENTRATIONS,  
WITH BLOCKING EFFECTS AND FORMATION OF COMPLEXES

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A new derivation of the relaxation strength for anelastic and dielectric relaxation of point defects is proposed, and the calculation is extended to cases not yet treated; the formulas reported in literature can be obtained as particular cases. The relaxation strength is expressed as the sum of the contributions of all the pairs of configurations which the defect can assume. The case of arbitrarily high concentrations with blocking, short range interactions and formation of defect complexes is also treated, and the formulas of the partial concentrations of the various defect configurations are provided. The proposed approach is useful if the formation of complexes and the short range interactions occur within independent cells or subsystems into which the crystal can be divided. Two specific cases are discussed as applications: the anelastic relaxation of a four-level tunnel system and of interstitial hydrogen in the hcp rare earths with the formation of H pairs.

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**P2-1**  
**STUDY OF THE DISLOCATIONS RELAXATION PEAKS IN EASY GLIDE DEFORMED SILVER  
SINGLE CRYSTAL HAVING DIFFERENT LENGTH TO WIDTH RATIO**

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**ABSTRACT**

The dislocation relaxation peaks in deformed single silver crystals of orientation  $\langle 110 \rangle$  i.e. easy glide, with length to width ratios of 8:1, 6.5:1, 4.9:1, 4:1 and 1:1 have been studied. These relaxation peaks have been measured at frequencies 5, 10 and 30 MHz. The results showed that a crystal with the ratio 8:1 produced highest dislocation relaxation peaks at relatively higher temperature. As the ratio decreased, the peak height decreased nearly linearly when the measurements were carried out at 5 MHz. At frequencies 10 and 30 MHz, the peak height fell dramatically for ratios less than 8:1, after which it decreased very little (nearly undetectable). The large dislocation relaxation peak height which occurred at relatively higher temperature in the sample of ratio 8:1 was attributed to the long dislocation loops formed during deformation. These loops seem to become shorter as the length to width ratio decreases.

**P2-2**  
**AMPLITUDE-DEPENDENT DAMPING AND ACOUSTOPLASTIC EFFECT DURING PLASTIC  
DEFORMATION OF ZINC SINGLE CRYSTALS**

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Amplitude dependences of the internal friction and of the acoustoplastic effect (APE) during plastic deformation of Zn single crystals have been investigated. *In situ* measurements have been made by the composite oscillator technique at frequency of about 100 kHz using automated computer-controlled setup. Rod-shaped samples oriented along [0001] axis were deformed by three-point bending. Ultrasonic standing wave has no shear component in the basal plane whereas static deformation is provided mainly by basal slip. The acoustoplastic effect (i.e. the decrease of the static load) is clearly registered at vibrational strain amplitudes  $\epsilon_0 > 10^{-5}$ . The rise of internal friction begins to appear at the amplitude approximately an order of magnitude lower. The results obtained can not be explained within the framework of both conventional superposition theories of APE and recent suggestion [1] on proportionality of the value of static stress decrease to the amplitude-dependent internal friction. The concept of local heating under influence of ultrasound is also concluded to be unable in explaining the mechanism of APE. Alternative mechanisms such as an easier dislocation glide through vibrating dislocation forest or an easier overcoming of vibrating point obstacles are discussed.

The changes in the amplitude-dependent decrement were not proportional to the changes in the amplitude-dependent modulus defect during deformation, unloading and reloading. This behaviour may be explained by a small (near 1 K) heating of the sample under the influence of ultrasonic vibrations.

[1] T. Ohgaku, N. Takeuchi, *Phys. Stat. Sol. (a)*, 105, 153 (1988).

## P2-3

### NON-LINEAR DYNAMICS OF DISLOCATIONS IN INTERNAL STRESS FIELDS AND MECHANICAL SPECTROSCOPY OF CRYSTALS

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The viscous glide of a dislocation in a non-linear potential field (the Peierls or point defect reliefs) under variable external loading is considered. Qualitatively new effects are initiated under the influence of the minor oscillating components of an external stress on the dislocation. A finite number of steady state oscillating regimes (dynamically equilibrium states - DES) are excited. Balance between energy accumulation due to external oscillating stress and energy dissipation due to viscous drag is obtained in a DES due to the non-linear feature of internal potentials. The number of dynamically equilibrium states is determined through the parameters of the "dislocation-barrier" system (the force-distance profile, viscous drag constant, etc.) and the form of the external oscillating stress component. The probability that dislocations break away from barriers, given rise to plasticity, increases due to transitions of the dislocation from one DES to another one due to superimposition of various weaker effects (step-by-step barrier surmounting).

The non-linear nature of the "dislocation-barrier" system can also cause the dislocation stochastic oscillations induced by the alternating external stress. Such oscillations are characterized by continuous frequency spectra. Excitation of stochastic oscillations also facilitates the process of overcoming obstacles by the dislocation.

The possibilities of the experimental discovery (the peculiarities of the mechanical spectroscopy) of non-linear dynamics of the dislocation and the peculiarities of the "dislocation-barrier" structure are discussed.

## P2-4

### ULTRASONIC EMISSION DURING THE CRACK PROPAGATION IN SOLIDS

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Sound radiation fields (the wave asymptotics of displacement velocity field and the stress field) generated by thin cracks propagated in infinity isotropic elastic medium are calculated. The brittle crack was replaced by the equivalent flat dislocation pile-up to interpret this crack as emission source. The pile-up includes continuously distributed dislocations with infinitesimal Burgers vectors. The dislocations perform nondiffusive climb in the direction of the crack beach broadening. Let the crack be the infinite band parallel to z-axis and laying in the  $XoZ$ -plane. Let also the crack width in the x-direction be known as the function of the time,  $2L(t)$ . If the speed of the crack propagation is sufficiently small,  $v = \partial L / \partial t \ll c_t$  ( $c_t$  is the velocity of shear sound waves) the dislocation density in the equivalent pile-up can be depicted by known quasistatic function  $\rho(x, y, t) = 2ax\delta(y)\{L^2(t) - x^2\}^{-1/2}$  ( $a = p(1-\sigma)/\mu$ ,  $p$  is external pressure applied to the crack,  $\mu$  is shear modulus,  $\sigma$  is Poisson ratio). Sound irradiation fields generated by crack at the distance  $R = (x^2 + y^2)^{1/2} \ll L$ , were calculated using dipole approximation similarly the generally accepted procedure developed in the theory of wave radiation. The above-mentioned crack emits the cylindric shear and pressure waves with amplitudes  $\propto \{\tilde{Q}(\varphi)/\sqrt{R}\} \partial^2 D(t)/\partial t^2$  (here  $D(t) = aL^2(t)$  is the dislocation dipole momentum per unit length of the crack,  $\tilde{Q}(\varphi)$  describes the angle distribution of sound radiation,  $\varphi$  is polar angle measured from positive direction of x-axis). Notice, that the intensity of sound radiation from a crack depends on the second time derivative of dipole momentum  $D$ . On the other hand, the magnitude of  $D$  is proportional to the free volume of crack. Directional diagrams  $\tilde{Q}(\varphi)$  are sufficiently different for shear and pressure components of total emission. This property can be used for the experimental controlling of crack acoustic emission which accompanies the fracture processes in real solids.

**P2-5**  
**DISLOCATION DAMPING AND ASSOCIATED MODULUS DEFECT IN COPPER CRYSTALS**  
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It is well known that the ultrasonic attenuation and modulus defect in pure copper crystals is due mainly to the dislocations in the crystal. Measurements of the dislocations ultrasonic damping and modulus defect in MHz-range, in deformed copper, showed that it is resulting of two contributing mechanisms: dislocation resonance (kink chain) and dislocation relaxation damping (Bordoni relaxation).

In his work criterias are presented to separate the contributions of each mechanism. Results obtained in  $\langle 110 \rangle$  and  $\langle 111 \rangle$  deformed copper single crystals revealed: a) the resonance damping depend on the frequency and the deformation; b) the Bordoni peak width is around twice a Debye peak.

**P2-6**  
**AMPLITUDE DEPENDENT INTERNAL FRICTION IN COPPER AT ULTRALOW TEMPERATURES**

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We have measured the amplitude dependence of the internal friction and of the modulus of polycrystalline copper at 300-400 Hz in the temperature range  $0.06 \text{ mK} \leq T \leq 900 \text{ mK}$  and in the maximum strain range  $3 \cdot 10^{-7} \leq \epsilon \leq 7 \cdot 10^{-4}$ . Our results show that at  $T < 300 \text{ mK}$  the internal friction  $Q^{-1} \propto \epsilon^n$  with  $n = 0.41 \pm 0.02$  for  $\epsilon > 3 \cdot 10^{-6}$  independent of temperature, e.g.  $Q^{-1} (T < 300 \text{ mK}) \approx 10^{-4}$  for  $\epsilon \approx 5 \cdot 10^{-6}$ . For lower strains  $Q^{-1}$  deviates from this dependence and tends to saturate; this deviation is larger the higher the temperature. The results will be discussed in terms of mechanical break-away and quantum-mechanical tunneling of dislocations. The temperature and strain dependence of the Young modulus resemble those of amorphous insulators as found for other polycrystalline metals.

## P2-7

### LUBRICATION MECHANISM AND THE RESTAURATION OF BORDONI RELAXATION IN Al 6N DEFORMED AT 7K

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Internal friction, dynamic modulus and plastic strain measurements were performed in high purity Al (6N) deformed at 7K, in order to study the lubrication mechanism in the dislocation mobility and its evolution with point defects recovery, at low frequencies ( $10^{-3}$  to 10Hz).

The plan consisted in isothermal stair-like annealings from 7K to 100K after 0.13% deformation by cycled torsion "in situ" at 7K.

Just after deformation, an important decrease in the dynamic modulus (softening) is observed. It confirms the existence of a lubrication process for the dislocation mobility assisted by vacancies produced during deformation.

Two different effects can be appreciated: an increase in the softening between 7 and 30K, and a partial recovery between 40 and 85K. These results can be interpreted in terms of a kink pair formation mechanism associated with the creation of an interstitial and a vacancy, as predicted by Quenet, EPFL Thesis 1992. The softening in the first range can be attributed to the increase in vacancy concentration, while the recovery in the second one, to the partial annihilation of vacancies due to the interstitial migration.

The complete restauration of Bordoni relaxation is produced at 100K when vacancies migrate along dislocation pipes.

## P2-8

### THE MODEL OF DISLOCATION AMPLITUDE-DEPENDENT INELASTICITY AND THE AMPLITUDE-FREQUENCY SPECTRA OF INTERNAL FRICTION IN CRYSTALS

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The experimental study of low-frequency amplitude-dependent internal friction (ADIF) and reversible dislocation strain in alkali-halides [1], fcc (aluminium) and hcp (zinc) crystal served as a basis for rheological model of amplitude and frequency dependent ADIF and Young modulus defect (YMD). This model was of unlocalized friction type and the concept of effective and internal stresses both contributing to ADIF was used. Attempt has been made to extend proposed model from quasistatic and low-frequency range to the ultrasonic one.

Comparison of calculated ADIF, YMD and stress-inelastic strain hysteretic loop, with experimental data on ADIF, YMD and experimentally observed in alkali-halides dislocation electric polarization was performed for ultrasonic range. It has been shown, that at ultrasonic frequencies the dependence of inelastic strain rate on effective stress does not obey the formalism [1], valid for macroscopic and low-frequency range. The conclusion was made, that at ultrasonic frequencies significant role in ADIF belongs to dislocations, athermally overcoming pinning points.

The rheological model was modified to cover wide frequency range. The qualitative microscopic model was proposed, in which dislocations overcome weak pinning points athermally, and pinning points with high interaction force in thermally activated way [2]. The restoring force due to reversible change of internal stress fields, i.e. dislocations interaction, was introduced to this unlocalized friction ADIF model. The simplifications and limitations of the model were also discussed.

1. S.N. Golyandin, S.B. Kustov. *Fiz. Tverd. Tela* (Sov. Phys. Sol. State), 1992, **34**, N12.

2. G. Gremaud. *J. de Phys.*, 1987, **48**, Coll. C8, C8-15.

**P2-9**  
**THE POWER SPECTRUM ASSOCIATED WITH A KINK CHAIN OSCILLATING IN A  
NONSTOKESIAN ATMOSPHERE OF PARAELASTIC INTERSTITIALS**

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The power spectrum of a geometric kink chain oscillating in an atmosphere of paraelastic interstitials which shows strong non-Stokesian viscosity regime in the strain amplitude dependent region is investigated numerically by utilizing Fast Fourier Transformation technique. Above a sharply defined threshold value of the strain amplitude, the odd-harmonic generation is observed only at the low temperature side of the dislocation relaxation peak. For high strain amplitudes, strong enhancement in the 9th and 11th harmonics, and the complete depression of the rest are seen very clearly during the computer simulations. Finally, the onset of quasi-chaotic kink oscillations are detected in the phase diagram while system strongly driven in the super-Snoek regime where the atmosphere tearing takes place.

**P2-10**  
**AMPLITUDE DEPENDENT INTERNAL FRICTION IN DILUTE COPPER ALUMINIUM ALLOYS  
WITH THREE KINDS OF PEAKS**

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Amplitude dependent internal friction was measured on dilute copper aluminium alloys using a torsion pendulum at about 1.6 Hz and between 300 K and 90 K. The concentration of aluminium was from 0.01 to 1 atomic per cent. After annealed and set in the pendulum, the specimen was twisted in situ to a small extent.

Three kinds of peaks were observed:

(1) Peaks in the amplitude dependence. The curve for the internal friction vs. amplitude had a maximum, a broad peak. The peak shifted to higher amplitudes, and the peak height increased, as the concentration increased.

(2) Peaks in the concentration dependence. It was shown that the amplitude dependent internal friction replotted against concentration also had a peak. The peak shifted to higher values of concentration, and the peak height increased, as the strain amplitude increased.

(3) Peaks in the temperature dependence. As the temperature decreased from 300 K to 90 K, the peak in the amplitude dependence shifted to higher amplitudes, and the peak height increased. It was shown that the internal friction replotted against temperature also had a peak. The peak shifted to lower temperatures, and the peak height increased, as the amplitude increased.

These results were interpreted in terms of the relaxation of dislocation in random solid solutions.

## P2-11

### WEAKENING AND ENHANCING EFFECTS OF SUBSTITUTIONAL ELEMENTS ON SNOEK-KE-KOSTER DAMPING

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The Snoek-Ke-Koster (SKK) damping peaks in Fe-N, Fe-P-N, Fe-P-La-N alloys and the commercially pure iron with and without mischmetal (RE) were studied. It was found that for the Fe-P-N alloy, there existed the following expressions:  $h_{sk} = (K_1 C_P^{-2/3} + K_2) h_s$ , and  $\tau = K_3 T C_P C_N^{-2/3} \exp(H/kT)$ , as well as the activity energy  $H$  and the peak temperature  $T_p$  decreased continuously as increasing of  $C_P$ ; here  $C_N$  and  $C_P$  were the concentrations of both solid solution N and P, respectively;  $h_{sk}$  and  $h_s$  were SKK peak height and Sonek peak one, and  $K_1$ ,  $K_2$ ,  $K_3$  were all constants. It was different from binary system that here the ratio  $h_{sk}/h_s$  is a function of  $C_P$ , but no a given value. These indicated that the P of solid solution in the alloy had a strong effect weakening SKK damping. Experiments showed that the RE in commercially pure iron also had the same weakening effect. It was seen, from a comparison between the Fe-P alloys with and without La, that the La in the alloy had an effect to enhance SKK damping. It was pointed out that SKK peak was caused by the string vibration of non-screw dislocations drawing Cottrell atmosphere of interstitial solution, and the reasons to produce the effects weakening and enhancing SKK damping were due to varied  $C_N$ ,  $C_P$  and/or the length of the dislocation segments by interactions between different substitutional elements and between substitutional and interstitials, and/or by substitutional atoms pinning the dislocations.

## P2-12

### PHOTOACOUSTIC PROPERTIES OF HgI<sub>2</sub> SINGLE CRYSTALS AT DIFFERENT LEVELS OF ULTRASOUND STRAIN AMPLITUDES

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Amplitude and time dependencies of ultrasonic damping and Young modulus of HgI<sub>2</sub> single crystals are investigated at room temperature by means of composite oscillator technique at frequency of longitudinal vibrations of about 100 kHz. It is found that acoustic parameters such as damping and resonant frequency of the sample (Young modulus defect) are strongly influenced by light. The spectral response of photoacoustic effect at small amplitudes (amplitude independent damping range) is appeared to be almost the same as that of photoconductivity. It is shown that the behavior of the effect at high amplitudes (amplitude dependent range) is quite different as compared with that one at small amplitudes. It is supposed that one can explain photoacoustic properties of HgI<sub>2</sub> by pinning of dislocations by centers which are created under light due to photoelectrons or holes.

**P2-13**  
**NONLINEAR MECHANICAL RELAXATION ASSOCIATED WITH DISLOCATION-POINT  
DEFECT INTERACTION**

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By nonlinear mechanical relaxation we mean that property of solids in virtue of which mechanical relaxation is accompanied by a nonlinear relationship between stress and strain, so that a strong amplitude effect is exhibited even under low stress levels. The amplitude effect is referred as anomalous when the relaxation strength decreases with increasing strain amplitude. This gives rise to the so-called anomalous internal friction which was observed for the first time by Kê in his study on the internal friction associated with the interaction between dislocation and point defect. It was found that an "amplitude peak" (versus strain amplitude) co-exists with the "temperature peak" (versus temperature).

This paper reviews the experimental results on the anomalous internal friction peaks observed in cold-worked and partially annealed aluminium containing Cu or Mg as point defects (solute atoms), with emphasis on the recent progress in the last ten years carried out in China. The fundamental problems tackled and answered include: (1) Whether the nonlinear anomalous internal friction is associated with an activation energy? (2) Where the solute atom atmosphere interacting with the dislocation is located? (3) What is the mode of diffusion of the solute atoms under the action of applied stress and the stress field of the dislocation? (4) Is the point defect constituting the atmosphere a single one or a complex one? (5) What is the origin and the mechanism of the anomalous internal friction peak?

In the course of intensive study, a series of seven internal friction peaks concerning dislocation-point defect interaction were observed. These peaks are related to each other. A unified "dislocation kink-point defect atmosphere" model with a "following, dragging, breaking" process was suggested for these seven internal friction peaks so that the specific explanations for each peak are self-consistent and support each other.

**P2-14**  
**COMPUTER SIMULATION OF THE GLIDE DISLOCATION MOVEMENT THROUGH  
ENSEMBLES OF LATTICE IMPERFECTIONS UNDER COMPLEX LOADING CONDITIONS**

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Some features peculiar to the glide dislocation movement through a forest of dislocation and a random ensemble of prismatic dislocation loops performing forced oscillations are analysed by computer simulation with respect to crystals having NaCl lattice and h.c.p. structure. The forest dislocation and dislocation loop ability to perform forced oscillation is shown to increase the transparency of corresponding ensembles. A critical value of dislocation loops forced oscillation amplitude, when the ensemble of dislocation loops becomes completely transparent for glide dislocation is determined. The existence of two amplitude ranges for the forest dislocation oscillation is established, with dissimilar weakening mechanisms occurring in each of them. Some regularities are discussed describing the dependence of the process characteristics on the forest structure and fine structure of inner stress fields.

## P2-15 SLIP LINES MOBILITY AND PLASTICITY OF CRYSTALS UNDER ALTERNATING STRESS LOADS

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The effect of the compressive and compressive-tensile stresses  $\sigma$  of different types and values ( $\sigma = (0.25 \text{ to } 100) \cdot \sigma_p$ ,  $\dot{\sigma} \approx 10^{-4}$  to  $10^3 \text{ MPa} \cdot \text{s}^{-1}$ ,  $\sigma_p$  is the yield stress) on both the mobility of slip lines (mean pathlength  $L(j)$ , mean number  $N(j)$ ,  $j$  is the ordinal number of loading), and the macroscopic work-hardening of very pure (P) and impure (I) NaCl single crystals has been studied as a function of temperature  $T=4.2$ , 77 and 298 K. The crystals were loaded by quasi-static, impulse, impact and ultrasonic (with different frequencies) treatments. It turned out that:

1. The dependences  $L(j, \sigma, \dot{\sigma}, T)$  change synchronously with  $N(j, \sigma, \dot{\sigma}, T)$  and usually exhibit a non-monotonic character with gradual fall of maxima and minima.
2. The attenuation of  $L$ ,  $N(j)$  is more prominent than that of individual dislocations, for low  $\sigma, \dot{\sigma}, T$ , compressive stresses, in I-NaCl and predeformed crystals. The stresses to start the dislocation unpinning ( $\sigma_s$ ) and multiplication ( $\sigma_M$ ) are strictly correlated in each case of above deformations.
3. In the slip lines there remain small dislocation loops that are, sometimes, revealed in a shallow etch pits ("the memory effect").
4. Such effects are observable in f.c.c. metals, semiconductors and solid  $^3\text{He}$ , etc. They are explained by the general model of dislocation motion, drag and multiplication by means of their cross-slip, motion of jogs and bowing the obstacles.

## P2-16 THE DISLOCATION-CRYSTAL DEFECTS INTERACTIONS AT EXTREMELY LOW AND HIGH TEMPERATURES

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The work deals with micro- and macrodeformation of NaCl single crystals in the temperature range  $T = (1.27 \cdot 10^{-3} \text{ to } 9.5 \cdot 10^{-1}) T_m$  ( $T_m$  is the melting point). The obtained results are compared with the published and unpublished data on dislocation mobility and microdeformation in normal and superconducting Zn and crystalline  $^4\text{He}$  at  $T \approx (6.45 \cdot 10^{-4} + 9.97 \cdot 10^{-1}) T_m$ .

The same character of microdeformation curves in solid  $^3\text{He}$  and  $^4\text{He}$ , and insulating metal, semiconductor and superconductor crystals is confirmed by the general conditions

$$\sigma_{st}(T, \dot{\sigma}) \approx \text{const } \sigma_p(T, \dot{\sigma}) \quad (*)$$

for these crystals, where  $\dot{\sigma}$  is the stress rate,  $\sigma_{st}$  is the stress to start the dislocation unpinning (or microdeformation) in the scales of observations by different methods: etch-pit technique (dislocation pathlengths  $l > 1 \mu\text{m}$  or deformations  $\epsilon \geq 10^{-3}$ ), electron microscope ( $l \approx 10^{-2} \mu\text{m}$ ), internal friction ( $\epsilon \geq 10^{-7}$ ),  $\sigma_p$  is the yield stress ( $\epsilon \approx 10^{-3}$ ).

Conditions (\*) confirm the unity of the micromechanisms of dislocation motion, multiplication and strain hardening and their natural interconnection with the Orowan bypassing the obstacles and cross-slip of dislocations in different classes of solids. These correlations contradict the mechanisms of quantum, viscous drag and inertia effects in the low-temperature deformation of different classes of crystals.

The data on extremely low-temperature (in the units of  $T_m$ ) deformation and structure of dislocations in solid helium ( $10^{-1}$ ), LiF ( $5 \cdot 10^{-4}$ ), NaCl ( $1.3 \cdot 10^{-3}$ ), Nb ( $5 \cdot 10^{-4}$ ), Mo ( $1.7 \cdot 10^{-4}$ ), Si ( $2.5 \cdot 10^{-3}$ ) and particularly in Cu ( $\geq 10^{-5}$ ) confirm "the model of microprecipitate hardening". It allows one to predict thermally activated dislocation motion with a finite but still vanishing activation energy down to the temperatures of disappearance of thermal expansion mismatch between precipitates and any matrix. In the last case like at  $T \leq T_m$  the lattice friction must be extremely low.

P2-17  
THE MICRO- AND MACROPLASTICITY OF SOLIDS AT LOW TEMPERATURES  
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The investigations were carried out on the effects of shear stress  $\tau$ , stress rate  $\dot{\tau}$  ( $10^{-4}$  to  $10^3$  MPa/sec) and temperature ( $T \approx 1.3$  to 298 K) on the deformation  $\gamma$  and the parameters of dislocation motion and multiplication (mean pathlength  $l$  and mean number  $n$  of mobile dislocations, mean pathlength  $L$  and width  $d$  of slip bands, mean density  $N$  of dislocations per slip band and density  $S$  of slip band sources) in plastic (P) and very plastic (VP) LiF and NaCl, P-KCl, KBr, KI and RbI single crystals. As usual the dependences  $l, n, \gamma, L, N, S, d$  ( $\tau, \dot{\tau}, T$ ) have two main stages from the general  $\gamma$ -shaped form. In P-crystals the sequence of stages corresponds to the so-called "parabolic" work-hardening at a single glide like in Cu single crystals with small particles of  $\text{SiO}_2$ , and in VP-crystals this sequence corresponds to the so-called "easy glide and linear work-hardening". At all  $T, \dot{\tau}$  and  $10^{-1}$  MPa  $\leq \tau \leq 64$  MPa the conditions  $\tau_M(\dot{\tau}, T, IC) \approx \text{const } \tau_p(\dot{\tau}, T, IC), \tau_{1,n,L,N,S,d}(\dot{\tau}, T, IC) \approx \text{const } \tau_\gamma(\dot{\tau}, T, IC)$  are fulfilled, where IC is the impurity state and concentration,  $\tau_M$  is the stress to start the multiplication of the first dislocation,  $\tau_p$  is the stress required to start the multiplication of  $p$  mobile dislocations,  $\tau_{1,n,\gamma,L,N,S,d}$  are the deforming stresses at  $l, n, \gamma, L, N, S, d = \text{const}$ . This fundamental property of micro- and macro-yield curves (in being similar to each other and to themselves at different points they occupy as the function of  $\dot{\tau}, T, IC$ ) confirms the unity of micromechanisms of dislocation motion, multiplication and macroscopic work-hardening and their close interconnection with cross-slip and climb of dislocations. The athermal bypass of the obstacles (impurity microprecipitates, microvoids, jogs, etc.) forms the distribution of the dislocation segment lengths and the sigmoidal shape of  $l, n, \gamma, L, N, S, d(\tau, \dot{\tau}, T)$ .

P2-18  
THE CORE STRUCTURE OF DISLOCATIONS AND THE PLASTICITY OF SOLIDS  
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The investigations were carried out on the effects of shear stress  $\tau$  ( $10^{-3}$  MPa  $\leq \tau \leq 64$  MPa), stress rate  $\dot{\tau}$  ( $10^{-4}$  to  $10^3$  MPa  $\cdot$  s $^{-1}$ ) and temperature  $T \approx (1.27 \cdot 10^3$  to  $9.5 \cdot 10^{-1}) T_m$  ( $T_m$  is the absolute melting point) on the dislocation motion and multiplication in different NaCl single crystals.

For all  $\tau, \dot{\tau}, T$ , thermal treatments of the samples (quenched or annealed ones, QA) and crystal predeformation at different  $T$  (PD) the conditions

$$(*) \quad \tau_s(\dot{\tau}, T, QA, PD) \approx \text{const } \tau_M(\dot{\tau}, T, QA, PD)$$

are fulfilled:  $\tau_s$  is the stress to start the dislocation unpinning,  $\tau_M$  is the stress to start the multiplication of dislocations. Correlations (\*) are strictly the same for insulating, semiconducting, metal and superconducting crystals, including the data from amplitude-dependent internal friction measurements (for solid He and He, metals, etc.). The different slopes of (\*) at  $\tau \leq 10^{-5}$  G and  $\tau > 10^{-5}$  G (G is the shear modulus) are concerned with the difficulties of dislocation multiplication at low stresses.

The numbers of cross-slip events  $q$  on screw dislocations and the probability  $p$  of the transition of dislocations from the cross-slip plane into one of the primary slip planes correlate with deformation stress in the same way for LiF, NaCl and Cu crystals at  $T=4.2$  to 300 K.

The last fact confirms conditions (\*) and their connection with cross-slips and the Orowan lowering of dislocations nearby the obstacles (nano-precipitates, jogs, etc.). These mechanisms are universal for all stages of micro- and macrodeformation and explain different temperature dependences of plastic flow and extremely low estimates of lattice friction ( $\tau_f \sim (10^{-7}$  to  $10^{-9})G$ ) in solids.

## THE EFFECT OF Cu AND Zr ON INTERNAL FRICTION OF AlMgZn ALLOY

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Internal friction (i.f.) and dynamic modulus (d.m.) of Al 2.0%Mg, 5.7%Zn, 0.008%Ti type alloys were investigated in as cast, extruded and rolled state. The measurements were made in an inverted torsional pendulum between room temperature (RT) and 600°C. Beside the master alloy (M marked above) the effect of additional Cu (c - 0.35%Cu; C - 1.4%Cu) and Zr (CZ - 1.4%Cu + 0.14%Zr) was also investigated.

Without any previous heat treatment in all the three as received state monotonously increasing i.f. and decreasing d.m. was observed above RT. Relaxational peak did not appear near the usual Ké-relaxation temperature range. The i.f. value (being about  $5 \cdot 10^{-4}$  at RT) increased to about  $200 \cdot 10^{-4}$  at 300°C for the cast and the extruded materials. The additional alloying elements increased the i.f. and this parameter was bigger for the cast materials. The Zr containing alloy do not follow the above trends. All the rolled alloys showed much bigger i.f. and lower relative d.m. at about 300°C (about  $2000 \cdot 10^{-4}$  and 0.35-0.5 respectively).

For all materials and starting from any conditions irreversible i.f. decreases and d.m. increases were detected due to recovery and recrystallization processes during the measurement. The temperature range of these transformations are (440-510)°C for the cast, (400-480)°C for the extruded and (290-320)°C for the rolled material. More alloying content increased the transformation temperature except for the Zr containing rolled alloy.

Heat treatments between 300 and 600°C reduced the i.f. in different amount and caused an appearance of relaxational peaks at about 400°C. The only unusual behaviour was found again for the Zr containing material which showed an anomalous amplitude dependence as well. From the above and other investigations too we can conclude that the CZ alloy might have a good superplastic character.

## P2-20

## DISLOCATION INTERNAL FRICTION, ACOUSTOPLASTIC EFFECT AND THE SIMILARITY LAW BETWEEN THE TEMPERATURE DEPENDENCES OF MICRO- AND MACRO-YIELD STRESSES IN SILICON BRONZE

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Polycrystalline silicon bronze has been investigated to obtain the comparison of acoustic properties with the mechanical ones. The experimental program included study of the peculiarities of internal friction (IF) and Young modulus during successive deformation of the samples of two compositions, simultaneous registration of amplitude dependences of IF and of the acoustoplastic effect (APE) directly during plastic flow of the samples, the measurements of the amplitude-dependent IF and of the macroscopic flow stress within the temperature range 6 - 300 K.

The behavior of IF and Young's modulus during successive deformation and annealing can be explained by the conventional concept of increase of number of depinned dislocations with strain. Some differences for two alloys were discussed. The threshold amplitude of APE can be clearly visible and this amplitude (in contrast to the data obtained earlier on various single crystals) is higher than that for amplitude-dependent IF. This disagreement can be explained by the difference between barriers for dislocations during micro- and macroyielding in polycrystals. Nevertheless, the similarity law between the temperature dependences of micro- and macroyield stresses (found recently in variety of single crystals [1]) holds in silicon bronze as well.

[1] A.B. Lebedev, S.B. Kustov, Phys. Stat. Sol. (a), 116, 645 (1989).

**P2-21**  
**THE PROBLEMS ON THE PEIERLS POTENTIAL IN FCC METALS**  
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Since the Bordoni peak had been found in FCC metals in 1949 and the kink pair formation (KPF) mechanism had been proposed for it in 1956, the Peierls potential has been investigated in various crystals. According to the recent aspect, the Bordoni peak around liquid nitrogen temperature in FCC metals is generally believed to be due to the KPF of dislocations to the Peierls potential and the  $\gamma$  peak above room temperature in BCC metals is virtually accepted to be due to the KPF of screw dislocations.

However, while the  $\gamma$  peak provides the reasonable magnitude for the Peierls stress  $\tau_p$  ( $\approx 10^{-2}\mu$ ,  $\mu$  is shear modulus) which agrees well with the estimated value from the low-temperature yield stress, the Bordoni peak provides rather large  $\tau_p$  ( $\approx 10^{-3}\mu$ ) compared with the suggested value from the plasticity of pure materials ( $\approx 10^{-5}\mu$ ).

This serious discrepancy has been tried to overcome by researchers just recently. From the point of view that the Bordoni peak should give the true Peierls potential, the idea of a lubrication mechanism is considered by Benoit and co-workers. The influence of inertial effects is also investigated by Nabarro, and Suzuki and Koizumi.

On the other hand, from the point of view that a new internal friction peak found at 11 K for 53 kHz in pure aluminum in 1989 may be the KPF peak, we have been able to derive a reasonably small  $\tau_p$  ( $\approx 3 \times 10^{-5}\mu$ ) compared with the plasticity measurement. In addition we have found that the 11 K peak satisfies all the features predicted from the KPF theory.

We would like to discuss the similarity and the difference in behaviors between the Bordoni peak and the 11 K peak quantitatively based on the KPF theory.

**P2-22**  
**ACCURATE DETERMINATION OF DISLOCATION-SOLUTE INTERACTION PARAMETERS IN**  
**Fe-C AND Fe-C-B OBTAINED FROM THE AMPLITUDE DEPENDENT INTERNAL FRICTION**

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The aim of the present work is accurate determination and comparative analysis of dislocation-impurity interaction parameters in Fe with low content of B and C. The investigation was made in temperature range of thermally assisted dislocation unpinning which is in agree with the internal friction model of Granato and Lucke (Appl. Phys. 52, 7136 /1981/). The binding energy of dislocation and point defect  $U_b$ , mechanical breakaway stress (at 0 K)  $\tau_m$ , up limit of thermally assisted internal friction range  $T_0$  and parameters of the force-distance law was estimated.

Fe-0.04 wt.% C:  $U_b=0.80 \pm 0.05$  eV,  $\tau_m/G=2.5 \cdot 10^{-3}$ ,  $T_0=405$  K

Fe-0.059 wt.% C-0.003 wt.% B:  $U_b=0.80 \pm 0.05$  eV,  $\tau_m/G=1.7 \cdot 10^{-3}$ ,  $T_0=415$  K

It was shown that B atoms are not more effective as a dislocation pinners compare with C and received value of  $U_b$  corresponds to the interaction between dislocations and carbon atoms in Fe. Details of the role of boron in solute-solution hardening of low-carbon steels are discussed.

P2-23

CRYSTAL WORK HARDENING DUE TO COMPLEX ENSEMBLES OF DISLOCATION FOREST  
AND POINT OBSTACLES (COMPUTER SIMULATION RESULTS)

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Computer simulation methods have been used to study the glide dislocation movement through a class of random composite ensembles of forest dislocations and point obstacles in crystals having NaCl lattice. It has been shown, that as a result of varying point obstacles relative concentration in the composite ensembles, various ensembles of point obstacles with different power becomes interchangeable in terms of the composite ensembles critical shear stress and some statistical characteristics of the process. The critical shear stresses for composite ensembles components have been stated not to be included additively in the total critical stress for composite ensembles. It has been shown that the sum of squares of critical stress values for the corresponding one-component ensembles can be applied to the calculation of total hardening for the composite ensembles under study.

P2-24

THE SIMILARITY LAW BETWEEN THE TEMPERATURE DEPENDENCES OF YIELD STRESS  
AND MICROYIELD STRESS EVALUATED FROM INTERNAL FRICTION

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Various definitions of microyield stress are considered. It is generally accepted that a precise registration of the onset of plastic flow in both macro- and microregions is practically impossible. In fact, macroscopic flow stress is defined as a stress  $\sigma_c$  corresponding to the certain value of irreversible plastic strain (as a rule, not less than  $10^{-4}$  -  $10^{-3}$ ). The same approach was applied to choose the microyield stress as a stress  $\sigma_c$  providing a constant level of reversible dislocation strain  $\epsilon_d$ . It is shown that one can obtain the temperature dependence  $\sigma_c(T)$  at very low  $\epsilon_d$  values ( $10^{-10}$  -  $10^{-8}$ ) from the data on amplitude-dependent internal friction (ADIF). It is shown also that the temperature dependences of microyield stresses defined in a different way (such as  $\sigma_w$  - the stress providing a constant value of energy loss in ADIF tests or  $\sigma_\delta$  - the one at a constant decrement of vibration) differ from  $\sigma_c(T)$  in relative units. The similarity law (i.e. the proportionality of the  $\sigma_c(T)$  and microyield stress) have been found for the  $\sigma_c(T)$  only [1]. This proportionality has been observed for a large variety of single and polycrystals of different crystal lattice, different impurity contents, orientation etc. The reasons for the similarity law are discussed within the framework of conventional thermally activated approach and an approach based on stress-induced plasticity. It follows from the similarity law that dislocation multiplication is an athermal process.

[1] A.B. Lebedev, S.B. Kustov, Phys. Stat. Sol. (a), 116, 645 (1989).

**P2-25**  
**SYSTEMATIC STUDY OF NON-LINEAR EFFECTS ASSOCIATED WITH SNOEK-KOSTER  
RELAXATION**

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Measurements in a torsion pendulum revealing the dependence of the internal friction on the amplitude of vibration in the temperature range of the Snoek-Köster relaxation, are systematically analysed in order to study different non-linear effects: amplitude dependent damping, peak temperature shift, as well as broadening and symmetry of the peak. The study was carried out before and after plastic deformation with monocrystals of different orientations and polycrystals (bamboo-like structure) of ultrapure niobium doped with different contents of oxygen (from 60 up to 1200 at.ppm).

Mechanical loss spectra are presented which allow the quantitative evaluation of the four non-linear effects mentioned above. For all the specimens it occurs, with higher or lower intensity, that the internal friction, the peak temperature and the HT-half-peak-width increase with the oscillating stress, whereas the LT-half-peak-width decreases. The four effects of amplitude dependence are more pronounced in specimens with low content of oxygen especially in the case of the polycrystal and of a  $\langle 110 \rangle$  monocrystal. Similar effects were already reported by other authors for other metals (fcc) and correlated with the Bordoni relaxation.

The results are discussed in the frame of the phenomena involving dislocation movement via kink pair generation in the presence of interstitial foreign atoms.

**P2-26**  
**INTERNAL FRICTION IN LAMELLAR EUTECTIC COMPOSITE Al-Al<sub>2</sub>Cu**

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Internal friction (IF) has been investigated within the temperature range from 6 to 300 K at frequency near 100 kHz with the help of computer controlled technique. Four types of IF experiments are presented: (i) the amplitude dependences of the logarithmic decrement measured at different temperatures; (ii) the temperature spectra of the amplitude-independent IF; (iii) the temperature dependences of the ultrasonic amplitude providing a constant level of the amplitude-dependent internal friction (ADIF); (iiii) simultaneous *in situ* measurements of the acoustoplastic effect (APE) and ADIF during room temperature plastic deformation. A comparison has been made between the data obtained in eutectic alloy and in Al-based solid solution.

The single-grain samples of eutectic alloy were obtained in the shape of rods about 2.5 mm in diameter and 80 mm length by Stepanov technique of shaped crystal growth. Aluminium and intermetallic layers approximately of the same width were interchanged with step 3.5  $\mu\text{m}$ .

Internal friction in eutectic composite may be attributed to dislocation microplasticity in Al-matrix. The results obtained at low temperatures are typical for Al as well as for other single and polycrystals of metals and alloys. Amplitude-dependent part of the decrement of vibrations is well approximated by a simple formula:  $\delta_h = A(T) \sigma_0^n$ , where  $\sigma_0$  is the stress amplitude,  $A(T)$  is a function of the temperature and  $n$  is a constant. The effect of small width of Al layers (less than 2  $\mu\text{m}$ ) is in the more pronounced strain ageing at  $T > 180$  K. Perhaps for this reason the mechanism of APE during active deformation is the dislocation multiplication (in contrast to increase of dislocation mobility in Al solid solutions).

**P2-27**

**COMPUTER SIMULATION OF DECORATION PEAK IN THE PRESENCE OF  
CONTINUOUS DRAG ACTING ON THE KINK CHAIN**

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The set of non-linear stiff differential equations which describes the kink chain oscillating in an atmosphere of smeared-out paraelastic foreign atoms and, in addition, decorated by a dragging point defect or its complexes, is solved numerically after having a novel scaling and renormalization procedure. The internal friction coefficient obtained indicates the existence of two separate relaxation peaks, the decoration peak and the parent peak which are directly related to the dragging localized point defects, and the smeared-out host foreign atoms, respectively. With slight modifications, the present computer modeling may be also utilized for the complete simulation of the transformation behavior of the Gamma peak into the Snoek-Koster peak upon the gradual condensation of interstitials at the sites of various kinks along the dislocation segment, in a distinct and/or random fashion.

**P2-28**

**LOW TEMPERATURE MECHANICAL LOSS SPECTROSCOPY OF LEAD 5N**

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The measurements of the internal friction as a function of the temperature in lead of purity 99.999 %, by a flexural vibration technique of thin clamped beams, show a relaxation peak at 37 K ( $\nu=660$  Hz) and at 32 K ( $\nu=249$  Hz). Measurements of internal friction using a forced inverted torsional pendulum have also revealed a relaxation peak at 20 K ( $\nu=1$  Hz) in lead 5N.

The calculation of the activation energy  $E_a$  gives 0.043 eV with a pre-exponential factor  $\tau_0$  of  $6.6 \cdot 10^8$  Hz. It seems that this relaxation is connected with a kink pair formation (KPF) mechanism on dislocations. But in comparison with the Bordoni relaxation's values in others fcc, we obtain much lower values for the activation energy and pre-exponential factor. To understand this low temperature relaxation in the lead 5N, it's necessary to compare these results with those obtained by a coupling method technique.

**P2-29**  
**NUMERIC SIMULATION OF ANELASTIC BEHAVIOUR AND MICRODEFORMATION DUE TO  
DISLOCATIONS**

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The attempts to calculate the mobility of dislocations in the atomic lattice carried out until this point have been concrete developpements that do not allow a general focus of the problem and are mainly dedicated to internal friction simulation. In this work, we introduce a general numeric simulation method that allows us to study the motion of the dislocations across the atomic lattice under different conditions of stress and temperature.

The energetic diagram of the dislocations is calculated, taking into account the length and internal stress distributions characteristic from the material. This way, we continuously calculate the statistic population of dislocations in each energetic valley and its evolution in time. The method allows us to follow the evolution of the distribution of dislocations induced by stress and temperature changes. Finally, the macroscopic deformation, associated to the motion of the dislocations, is obtained.

This calculation method allows us to simulate different experimental conditions, getting the micro-creep and elastic after-effect curves as well as the microdeformation obtained at a constant stress during heating. The internal friction spectra versus temperature have been simulated in a similar way.

**P2-30**  
**ON THE POSSIBILITY OF THE RESONANT-TYPE ABSORPTION OF OSCILLATORY ENERGY  
IN INHOMOGENEOUS SOLIDS**

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In our experiments we observed the sharp internal friction peaks, accompanied by the leaps of the resonant frequency of three component oscillator during deformation of the samples of silicon bronzes, Al-Mn and Cu-Al-Ni alloys. In the vicinity of peaks resonance curves of oscillator had two closely-spaced maxima and the leaps of resonant frequency corresponded to the transition from one maximum to another during their smooth change. Some interconnections between structural parameters of the investigated materials and characteristics of the anomalies were found.

In a number of experimental works, where elastic and inelastic characteristics of crystalline solids were measured as a function of temperature or frequency, the resonance-type dispersion of these characteristics was observed. This last looked like sharp absorption peaks, accompanied by the abrupt (often step-like) dynamic elastic modulus changes. In some works the splitting of the resonant frequency of the vibrational system was observed in the certain temperature ranges. The attempts were made to explain these anomalies by several different reasons.

Based on our own and reviewed works, we suppose, that the observed anomalies are due to the resonant interactions of the elastic wave with structural inhomogeneities in solids, and the resonant absorption of oscillatory energy is caused by appearance of additional 'internal' degrees of freedom of the vibrational system connected with these inhomogeneities. We assume that different types of volume or planar defects of crystalline solids can act as such inhomogeneities.

## P2-31

### AMPLITUDE DEPENDENT INTERNAL FRICTION AND YOUNG'S MODULUS DEFECT DURING PSEUDOELASTIC DEFORMATION OF Cu-Al-Ni SINGLE CRYSTALS

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The amplitude-dependent internal friction (IF) and Young's modulus defect (YMD) of Cu-14.0Al-3.8Ni (mass %) single crystals ( $M_s=263$  K) have been studied *in situ* during stress-induced pseudoelastic transformation at room temperature. Three component resonant oscillator technique was used for measurements during three point bending of the samples. The strain amplitude range covered  $10^{-7}$ - $3 \cdot 10^{-5}$  at frequencies near 100 kHz.

The IF maximum accompanied by minimum of resonant frequency was observed during pseudoelastic deformation of samples when measurements were taken at the constant value of strain amplitude within amplitude-independent range.

The amplitude dependencies of IF and YMD were measured at different experimental points during deformation. The significant reversible rise of IF and YMD without marked hysteresis occurred when the amplitude exceeded  $3 \cdot 10^{-6}$ . Only slight changes of amplitude-dependent IF were observed with the significant increase of amplitude-independent IF during deformation of two-phase samples. It has been found, that ultrasonic oscillations had no influence on the stress-strain behavior of the samples in the whole investigated amplitude range. There lies the discrepancy between transformation pseudoelasticity and dislocation plasticity. In the latter case the quasistatic flow stress can decrease under superposition of oscillatory stress in amplitude-dependent range (so called acoustoplastic or Blaha effect). One of the possible reasons of the fact is that amplitude dependence of IF and YMD is due to defects, which are not responsible for pseudoelastic deformation.

## P2-32

### AN ULTRASONIC STUDY ON BEHAVIOUR OF DISLOCATIONS IN ALUMINIUM DEFORMING AT HIGH RATES OF STRAIN

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It is widely known that the dynamic flow stress of metals shows a steep rise at strain rates above about 5000/sec. This phenomenon has long been interpreted in terms of the transition in the rate controlling mechanism of dislocation motion from the thermally assisted cutting of forest dislocations at lower strain rates to the viscous drag of phonon origin at very high strain rates. Recently, an interpretation in terms of the strain rate dependency of the evolution of dislocation structure during deformation has been presented. The former interpretation is based upon the effect of the instantaneous strain rate while the latter puts emphasis upon the role of the strain rate history.

In this work in order to obtain experimental information on the behaviour of dislocations under dynamic plastic deformation, time-resolved measurements of the attenuation of the ultrasonic pulse superimposed upon the dynamic deformation have been made for high purity polycrystalline aluminium at strain rates up to 12000/sec.

Theoretical analysis based upon continuum mechanics shows that the attenuation under plastic deformation is proportional to the dependency of the flow stress upon the *instantaneous strain rate*. A close correlation is observed between the *instantaneous strain rate dependency* of the flow stress evaluated from the results of the above attenuation measurements and the *strain rate dependency* of the flow stress obtained from the direct flow stress measurements. This means that the flow stress depends much more upon the *instantaneous strain rate* than the *strain rate history*. The ultrasonic results are also analysed using a kinetic model of dislocation motion in which the velocity of mobile dislocations is controlled both by the thermally assisted cutting of the forest dislocations and the viscous drag. The results indicate that the role of the viscous drag becomes dominant at strain rates above about 5000/sec. The results of the above two analyses of the ultrasonic data support the interpretation that the steep rise in the flow stress is due to the transition in the rate controlling mechanism of mobile dislocations.

P2-33

OSCILLATORY SPECTRUM OF DISLOCATION INTERACTING WITH DISORDERED LOCAL DEFECTS

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Oscillatory spectra of dislocations elastically interacting with disordered local defects (impurities) are studied. The problem is considered involving more general assumptions as compared to those used in known models treating impurities as points of stiff dislocation pinning. It is assumed that an oscillating dislocation is influenced by the Peierls relief and local defects randomly distributed over the crystal volume and in the region of the dislocation core. The local defects in the dislocation core create potential wells of finite depth, which are localized at atomic distances. In the framework of the model, the study of free oscillations of the dislocation leads to investigation of the eigenvalues of the single-particle Schroedinger equation with a random Poisson type potential.

General properties of the frequency spectrum are found and analytical expressions are derived for the spectral density in the limiting cases of strong and weak impurities. It is shown that the main result of the impurity influence on dislocation oscillations is the gap appearing in the frequency spectrum and the spectral density maximum near it. The dependence of the spectral density on impurity concentration and strength is analysed. The results are generalized for the case when several kinds of impurities are present in the crystal. Particular cases are analysed in more detail: (i) all defects are weak or strong; (ii) some defects are weak, and some ones are strong.

P2-34

PECULIARITIES OF AMPLITUDE-INDEPENDENT DISLOCATION INTERNAL FRICTION IN DISORDERED SOLID SOLUTION

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A generalization of the well known Granato-Lucke theory of amplitude-independent internal friction caused by viscous drag of sound excited dislocations is proposed. It is assumed that an oscillating dislocation is influenced by the Peierls relief and by impurities randomly distributed over the crystal volume and in the dislocation core. It is assumed that the impurities positioned within the dislocation core create potential wells of finite depth. The impurities exterior to the dislocation core produce a random potential relief that is determined by the superposition of their elastic fields. The general relationship which related the complex dislocation compliance to the averaged Green function of stationary oscillations of an underdamped dislocation segment is derived. A statistical averaging is performed within the framework of phase formalism, and rigorous mathematical relations are derived which permit the dislocation compliance to be determined for the case of disordered impurities. Explicit expressions for decrement and modulus defect are found and analysed in a practically important region of parameters, where the frequency dependence of decrement is of the Debye type. The quantities to be measured are found on impurity subsystem parameters, namely, impurity concentration and strength. It is shown that the decrement and the modulus defect are in exponential dependence on point defect concentration but this dependence is move weak than that predicted by the Granato-Lucke theory. On the general case a knowledge of only the average dislocation density is insufficient to determine the drag coefficient of dislocations but rather more detailed data on length distribution of dislocation segments are required.

P2-35

MIGRATION MECHANISM OF SELF-INTERSTITIAL ATOM IN Mo AFTER LOW-TEMPERATURE IRRADIATION: II DISLOCATION PINNING

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For the migration mechanism of self-interstitial atom (SIA) in Mo after low-temperature irradiation, in Part I in this conference, we found the following: For a very low Frenkel-pair concentration ( $C_{FP}$ ) around  $10^{-2}$  ppm, all the free-SIA's can migrate three-dimensionally with rotation. With increasing  $C_{FP}$ , part of SIA's come to migrate without rotation leading to a decrease in the fractional ratio of SIA's which can migrate with rotation. These facts would be responsible for the somewhat complicated recovery behavior of SIA's reported in the resistivity measurements as well as for the broad pinning caused by the migration of SIA's at around 40K (the 40K-pinning, hereafter). To clarify this issue, in Part II here, we carried out careful measurements for the 40K-pinning in Mo in the wide  $C_{FP}$  range between  $10^{-4}$  and 10 ppm by means of 20 MeV proton irradiation at 5 K. For very low  $C_{FP}$  below  $10^{-2}$  ppm, the 40K-pinning rapidly moves to higher temperatures with decreasing  $C_{FP}$ , where we surmise that some effects of impurities come in. For  $C_{FP}$  above  $10^{-2}$  ppm, the 40K-pinning shifts to lower temperatures in proportion to  $\log(C_{FP})$  as a whole. This general  $C_{FP}$ -dependence can be explained by the reasonable model for dislocation pinning due to the migration of free defects assuming the activation energy of 83 meV, that is reported for free-SIA's in the resistivity measurements. Further, a close investigation of the 40K-pinning suggests that the 40K-pinning observed for  $C_{FP}$  above  $10^{-2}$  ppm is composed of two constituents, the lower and the higher temperature pinnings (the LT- and HT-pinnings, hereafter): The LT-pinning is predominant at low  $C_{FP}$ , and shows relatively small temperature shift with varying  $C_{FP}$ . On the other hand, with increasing  $C_{FP}$ , the HT-pinning becomes predominant and largely shifts to lower temperatures. From the  $C_{FP}$  dependence of the pinning rate, we surmise that the LT- and HT-pinnings correspond with the migration of SIA's with rotation and that without rotation, respective! .

P2-36

EVIDENCE FOR DISLOCATION MULTIPLICATION DURING LOW TEMPERATURE THERMAL CYCLING OF M.M.C. SHOWN BY THE ULTRASONIC-LOW FREQUENCY STRESS COUPLING TECHNIQUE

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In metal matrix composites the difference in coefficients of thermal expansion of matrix and particles has been claimed by many authors to be responsible for the dislocation punching from the particle/matrix interface on cooling composite materials. Therefore, an experimental study has been undertaken by means of the ultrasonic-low frequency stress coupling technique in order to support this assumption.

This study is carried out on an Al / 12 vol% SiC composite. The attenuation changes ( $\Delta\alpha$ ) are measured, in isothermal conditions, during the application of a low amplitude slowly varying stress. This experiment is repeated at equally spaced temperatures ranging from 280K to 80K, successively on cooling and on heating the specimen. The amplitude of the attenuation change  $\Delta\alpha_c$  varies strongly with temperature by exhibiting a marked maximum around 240K. Moreover, whatever the temperature, on heating this amplitude is larger than that on cooling. These phenomena are discussed in terms of dislocation-point defect interaction and dislocation punching out from the particle/matrix interface during the low temperature thermal cycle.

P2-37

SENSIVITY TO DYNAMIC STRAIN AGING IN C-Mn STEELS

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In C-Mn steels and associated welds the Dynamic Strain Aging (DSA) phenomenon induces an increase of the ultimate tensile strength associated with loss in ductility (elongation and fracture toughness) in the 100-200°C temperature range. In the Heat Affected Zones of welds, the sensitivity to DSA, which is microstructure dependent, can not be directly determined. Therefore, in order to study the influence of microstructure on DSA, internal friction and tensile test results on simulated HAZ microstructures have been performed and compared.

Tensile tests (between 20 and 300°C) and internal friction experiments (at 1Hz between - 20 and 600°C) were performed on materials with different sensitivity to DSA: Manual Metal Arc Weld (0.49%C, 90 ppm N), normalized aluminium killed A 42 steel (0.14%C, 82 ppm N) and silicium semi-killed A 48 steel (0.198%C, 83 ppm N) with various heat treatments (normalized or austenized at 1250°C or 1050°C and water quenched). Before testing, the materials were aged at room temperature during several months.

For all these different microstructures, the intensity of the DSA, evaluated by the ductility loss, is proportional to the Snoek peak height. The microstructures obtained after water quenching are the less sensitive to DSA. This lower sensitivity is attributed to the high dislocation density induced by quenching and revealed by the cold work peak height: dislocations trap N and C atoms hence decreasingly the amount of free interstitial C and N atoms in the lattice, which are involved in the DSA phenomenon.

All these results confirm that even for industrial materials the height of the Snoek peak (determined in one test) is a good indicator of the DSA sensibility.

P2-38

DISLOCTRON DYNAMICS - A NEW APPROACH TO THE ANELASTIC INTERACTION BETWEEN DISLOCATION LINE AND POINT DEFECTS

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Discrete calculation showed that the main contribution of the anelastic interaction between mobile point defects and the dislocation is due to the defects in the core region. A model of chain of disloctrons, each of them is a least separable segment of dislocation line, was put forward. Under a discrete elastic approach, the fundamental parameters associated with a disloctron including self energy, interaction rigidity between two neighboring disloctrons, P-N stress and dynamic rigidity due to the mobile point defects near core of disloctron etc., were calculated numerically. Their values and even signs vary dramatically with the position of the disloctron. The chain of disloctrons satisfies an extended Frenkel-Kantlova equation. The static multi-soliton solution of it could be obtained numerically by using a relaxation approach. Finally, the complex amplitude of oscillation of the disloctrons and internal friction were obtained by using Gauss elimination method for sparse matrix.

As an example, the anelastical interaction between the vacancies and dislocation line in a fictitious elastic isotropic orthogonal lattice was analysed. The fundamental parameters of the disloctron, the width of the kink, the internal friction as the function of frequencies, the concentration of vacancies, the slope of the dislocation line with respect to the P-N valley and inner stress were obtained. The P-N stress and the width of the geometric kink are dependent on the concentration of vacancies. Furthermore, the width of the kink decreases as the density of the kinks and the inner stress increase. The results of the internal friction were quite different from that predicated by string or kink models. An increasing internal friction peak appears in an "annealed specimen" according to the increase of the concentration of vacancies. The inner stress or a few of geometric kinks widen this peak. Sometimes double peaks or a high background were observed.

P2-39

THERMALLY ACTIVATED RECOVERY OF POLYCRYSTALLINE CADMIUM DEFORMED BY ANISOTROPIC THERMAL EXPANSION

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The recovery of polycrystalline cadmium plates, after being deformed by internal stresses due to anisotropic thermal expansion during cooling, has been investigated by measuring the increase in the vibration frequency of the plate with time, and varying the temperature as a parameter. The recovery of frequency with time showed to follow an exponential law with a characteristic time of the order of few hours, thus reaching a limiting frequency after sufficiently long time. This limiting frequency is found to increase with the increase of the annealing temperature. The activation energy controlling the recovery process is about 0.12 eV. The recovery is explained in terms of diffusion of self interstitials produced during the deformation by the intersection of basal dislocations with the forest dislocations.

P2-40

VARIATION OF INTERNAL FRICTION AND ULTRASONIC ATTENUATION IN ALUMINIUM DURING THE EARLY STAGE OF FATIGUE LOADING

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Variation of internal friction  $Q^{-1}$  and ultrasonic attenuation  $\Delta\alpha$  versus cyclic number  $N$  of push-pull fatigue loading with various stress amplitudes  $\sigma$  for 99.999wt% Al polycrystalline specimens were measured. The corresponding dislocation configuration in the specimen was observed by transmission electron microscopy.

Experimental results showed that the dislocation configuration corresponding to the  $\Delta\alpha$  maximum on the  $\Delta\alpha - N$  curve is consisted of tangle bands accompanying with single dislocation segments located between the tangle bands. Meanwhile, the  $\sigma$  maximum on the  $\sigma - N$  curve corresponds to an incomplete dislocation cell structure.

The change of the internal friction  $Q^{-1}$  versus cyclic number  $N$  was found to be synchronous with that of the stress  $\sigma$ .

# ATHERMIC AND THERMOACTIVATED MOTION OF DISLOCATIONS IN ALKALI-HALIDS CRYSTALS UNDER HIGH HYDROSTATIC PRESSURE

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Both unactivated and thermoactivated movement of individual edge dislocations in KCl and NaCl single crystals under stresses  $\sigma < \sigma_{yield}$  at pressure up to 700 MPa were studied. For the investigation of this movement the specimens were subjected to stress pulse of the triangle or trapezoidal shape. The dislocations path length  $\ell$  as a function of applied stress  $\sigma$ , pressure P, temperature T, stress rate  $d\sigma/dt$  and time  $t$  (for plateau of stress pulse) was determined in experiments.

It was found that dislocations motion is of different nature at different stage of pulse loading: it is unactivated on a front of pulse load increase and thermoactivated on a plateau of pulse.

For the unactivated motion of dislocations it was found that  $\ell_f$  depend not only on  $\sigma$ , T and P, but on the stress rate  $d\sigma/dt$  too. It will be noted that  $\ell_f$  increases with decreasing T and increasing P according to the temperature and pressure dependence of the shear modulus G. The corresponding dislocations velocity  $v_f$  in this case, is a function only of the loading rate  $d\sigma/dt$ .

For the thermoactivated motion of dislocations the stress dependence of the velocity of dislocations in terms of  $\ln v_p - \sigma$  for varies T and P were obtained. It was found that  $v_p$  at  $\sigma = \text{const}$  the rapidly decrease with P and at  $v_p = \text{const}$  the applied stress  $\sigma$  increase with P according to  $G(P)^{3/2}$ . It was discovered that the velocity  $v_p$  of dislocations in KCl crystals has a peak at stress  $\sigma \sim 0.1 \sigma_{yield}$  after which the  $v_p$  decrease with increasing  $\sigma$  and then  $v_p$  increase with stress at  $\sigma > 0.5 \sigma_{yield}$ .

The experimental results are discussed from the point of view of point defects clusters reconstruction under applied stresses.

A SIMPLIFIED THEORY FOR THE LOW-FREQUENCY DRAGGING AND BREAKING-AWAY  
PROCESS OF MOVING DISLOCATION KINKS

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Recent systematical experimental results of anomalous internal friction in aluminium solid solutions observed by T. S. Kê et al. can not be explained by conventional dislocation damping theories, e.g., G-L depinning model and linear dragging model. In this paper, a new concept of dynamical breaking-away of dislocation from bound point defects is proposed. Starting from the lagging of the diffusional drift of the bound point defects behind the dislocations (kinks), the criterion of the immobility of the bound point defect was introduced as  $kT/CD \ll 1/\omega$ , where  $k$  is the Boltzmann constant,  $T$  the temperature,  $C$  the harmonic constant of the interaction between dislocation (kink) and point defect,  $D$  the diffusion constant of the point defect and  $\omega$  the frequency of applied stress. The linear relationship between the deviation  $\Delta x$  from the equilibrium spacing of the interacting configuration {dislocation(kink)-point defects} and the rate of motion  $u$  of the dislocation (kink) was derived as  $\Delta x = (kT/CD)u$ . Based on these, a simplified theory for the low-frequency dragging and breaking-away of dislocation kink was established through a simple description on the process of the static dissociation(depinning), overall motion (dragging) and dynamical dissociation (breaking away) of the {dislocation (kink)-point defects} configuration under the action of the low-frequency applied stress. The internal friction and the change of elastic modulus, especially their variation with stress amplitude and temperature were calculated and compared with relevant experimental results.

**P3-1**  
**ULTRASONIC DISPERSION IN PHASE TRANSITION REGION IN FERROELECTRIC MATERIALS**

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The results of experimental investigations of critical ultrasonic behavior near the phase transitions in ferroelectric materials are presented in this communication. Three main cases in the vicinity of phase transition temperature  $T_c$  of ultrasonic wave behavior are discussed: i)  $\omega\tau < 1$  ( $\omega$ -the angular frequency of ultrasonic wave,  $\tau$ -the order parameter relaxation time), ii)  $\omega\tau > 1$ , iii) the intermediate case  $\omega\tau \sim 1$ . In our ultrasonic frequency range 10-100 MHz the condition  $\omega\tau < 1$  was realized in the most of our investigated uniaxial ferroelectrics  $\text{Sn}_2\text{P}_2\text{S}_6$ ,  $\text{PbHPO}_4$ , DGN. In this case usually the stepwise downward jump was observed when temperature was approaching  $T_c$  from paraelectric phase for longitudinal ultrasonic modes propagating perpendicular to the polar axis. The temperature dependence of ultrasonic wave velocity in the ferroelectric phase could be described using phenomenological Landau theory. The ultrasonic velocity dispersion is impossible to observe even at the Brillouin experiments because it must arise in extremely narrow temperature range. The condition  $\omega\tau > 1$  was fulfilled in the crystals of DMAAS because of comparatively low polarization relaxation time. In this crystal we had not observed any critical slowing down of ultrasonic velocity as well as the frequency dependence of ultrasonic velocity. In the ferroelectric phase the ultrasonic velocity have the additional part proportional to the square of the polarization. The most interesting behavior of ultrasonic velocity was observed in the ferroelectric DDSP single crystals, when the longitudinal ultrasonic wave propagated along the [100] direction. The frequency dependent velocity dips and corresponding to the attenuation maxima were observed in the ferroelectric phase. The ultrasonic attenuation maxima appearing at  $\omega\tau = 1$  had shifted to lower temperatures when frequency was increased. The velocity dip near phase transition was strongly dependent on frequency and almost disappeared at 90 MHz frequency.

**P3-2**  
**EFFECT OF THERMAL AND THERMO-MECHANICAL CYCLING ON THE PHASE TRANSFORMATIONS IN NiTi AND  $\text{Ni}_{47}\text{Ti}_{50}\text{Co}_3$  SHAPE MEMORY ALLOYS**

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Microstructural evolutions of two types of shape memory alloys (S.M.A.), NiTi and  $\text{Ni}_{47}\text{Ti}_{50}\text{Co}_3$  are studied as a function of temperature during heating or cooling between - 80 and + 100°C.

Experiments consist in internal friction (low frequency : 1 Hz.) and differential scanning calorimetry (D.S.C.) measurements. The aim of this work is to determine if the phase transformations sequence :

$A$  (Austenite B2)  $\leftrightarrow R$  (Rhomboidal)  $\leftrightarrow M$  (Martensite B 19')

effectively occurs in these alloys and if these transformations are reversible. This study shows that the structural evolution depends on various parameters such as the chemical composition of alloys, single thermal cycling or combined thermo-mechanical cycling.

The main results concerning the influence of these parameters are the following :

- the R phase is always detected, on cooling, whatever the material is, but with more difficulty in NiTi alloys;
- thermal cycling (D.S.C. experiments) implies an austenitic phase stabilization leading to a shift of the martensitic transformation towards low temperatures while the R phase temperature is maintained fixed;
- compared to this effect, internal friction experiments, likened to thermo-mechanical cycling, give rise to an improvement of the austenitic phase stabilization proved by an additional shift of the martensitic phase transformation temperature;
- the most interesting result of this thermo-mechanical stabilization consists in a very clear separation of the phase transformations  $M \rightarrow R \rightarrow A$  on heating, in  $\text{Ni}_{47}\text{Ti}_{50}\text{Co}_3$  alloys. Such an effect is not observed in NiTi alloys, nevertheless thermo-mechanical cycling allows the distinction  $A \rightarrow R \rightarrow M$  on cooling.

Small alternative strains, inherent to an internal friction experiment, lead to noticeable structural modifications. The observed phenomena are explained on the basis of a progressive locking of lattice defects in the austenitic phase. The stabilization effects, by thermal or thermo-mechanical cycling, are compared with the influence of preliminary annealings.

**P3-3**  
**MARTENSITIC TRANSFORMATION IN Ni-Ti ALLOY STUDIED BY ISOTHERMAL MECHANICAL SPECTROMETRY**

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A 50,5 % at. Ti - 49,5 % at. Ni shape memory alloy was studied between 270 K and 400 K by isothermal mechanical spectrometry in a large frequency range ( $10^{-4}$  Hz - 150 Hz).

Results presented here are very different than those obtained by different authors using anisothermal method. So, at high frequency, internal friction decreases directly from the martensitic level to the austenitic one, without the large damping associated with a  $T$  effect.

Low frequency measurements show a thermally activated effect present only in the martensitic phase and decreasing with the time at constant temperature. This effect is discussed with segment dislocation motion inducing a variant interface moving.

**P3-4**  
**THE INFLUENCE OF CHEMICAL COMPOSITION AND TREATMENT REGIMES ON TEMPERATURE DEPENDENT INTERNAL FRICTION OF INVARS IN THE RANGE OF PHASE TRANSFORMATION**

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Temperature and amplitude dependent internal friction (IF) and modulus of elasticity (ME) of commercial polycrystalline alloys Fe-(24÷31,5)Ni and Fe-25Ni-5Mo with different carbon content (0,002 - 0.09 wt. %C) were investigated after different regimes of cooling and annealing.

The influence of chemical composition and heat treatment regimes of invars on carbon redistribution in FCC lattice, on the athermal  $M_A$  and isothermal  $M_I$  character of  $\gamma$ - $\alpha$  transformation and subsequent stages of martensite decay will be discussed with the help of IF and ME measurements. The increase of carbon content leads to the decrease of  $M_I$  temperature (from +5 to -70°C),  $M_A$  temperature increases. Only athermal "burning" transformation takes place in alloys with 0.37 wt.%C. Computer simulation was used for subdividing components of complex peaks to simple partial effects.

**P3-5**  
**INFLUENCE OF AGEING ON THE REVERSIBLE MARTENSITIC TRANSFORMATION IN Cu-Zn-Al ALLOYS**

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The methods of internal friction and electrical resistance were applied to study the influence of the process of ageing of the martensite in two Cu-Zn-Al alloys on the form and variations of the characteristic temperatures of the reversible martensitic transformation. Analysis of the internal friction curves for Cu-(12.7% at.)Zn-(18.0% at.)Al after various martensite ageing times showed that in the initial period of the martensite stabilization process there is an increase in the characteristic temperatures of the martensite-matrix phase transition, and vice versa. Increase in ageing temperature leads to hindering the course of the reversible martensitic transformation. Relations obtained showed that the martensite stabilization is a thermally activated process in which a fundamental role is played by the quenched-in vacancies formed during quenching from the high temperature of the  $\beta$  phase. The activation energy of the martensite stabilization process for initial stage a value  $E=(0.73\pm 0.01)\text{eV}$  was determined when making use of the chemical reactions rate equation.

For the second tested alloy (Cu-(12.4% at.)Zn-(17.2% at.)Al), basing on the analysis of isochronous and isothermal curves of resistivity determined in the temperature range from 273K to 343K, two elementary processes A and B were distinguished. Process A of activation energy  $E_A^M=(0.69\pm 0.02)\text{eV}$  corresponds to the disappearance of bivacancies, while B with activation energy  $E_B^M=(0.84\pm 0.02)\text{eV}$  corresponds to the disappearance of monovacancies.

**P3-6**  
**INTERNAL FRICTION DURING  $\lambda \rightleftharpoons \epsilon$  TRANSFORMATION OF Fe-Mn ALLOY**

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The internal friction and relative modulus during the process of  $\gamma \rightleftharpoons \epsilon$  transformation in Fe-18.8Mn alloy were measured by a vacuum torsion pendulum. The internal friction during heating, cooling and during isothermal aging was each and all measured. The athermal rate  $\dot{T}$ , the frequencies of measurement were 0.5K/min to 3.5K/min and 0.6Hz to 2.0Hz respectively. Two components of IF, athermal and isothermal, and two types of IF peak corresponding to  $\gamma \rightleftharpoons \epsilon$  transformation, athermal and static (stable) are observed during heating and cooling. The athermal IF shows a viscoelastic feature and increases with an increasing of  $(\dot{T}/\omega)^n$  but the isothermal IF ( $Q^{-1} - t$  curves at a certain temperature) increases with decreasing  $\omega$ . A phenomenological interaction model is proposed to explain the characteristics of athermal IF and isothermal IF.

### P3-7

## ULTRASONIC BEHAVIOUR OF DEUTERATED BETAIN ARSENATE CRYSTALS WITH TWO LATTICE INSTABILITIES

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Deuterated betaine arsenate (DBA) is an example of crystals with two lattice instabilities. With the deuterium content more than 80% there are two lattice instabilities in these crystals at  $T_{c2}$  (structural instability) and  $T_{c3}$  (ferroelectric instability). At 85% deuterium  $T_{c2}=161.5K$  and  $T_{c3}=145K$ . The Landau thermodynamic potential with two order parameters was analysed and it was shown that the physical properties in the phase transition regions depended critically on the coupling of two order parameters. Temperature dependences of attenuation and velocity of longitudinal ultrasonic waves with frequencies 15 and 30 MHz were measured in DBA 83 and DBA 85. At the  $T_{c2}$  point a step-like behaviour of velocity and attenuation peaks were observed, as for the  $T_{c3}$  point a positive jump of velocity with no attenuation peaks were found. The experimental results on ultrasonic properties and the data of dielectric measurements are discussed in the framework of the theory of crystals with two lattice instabilities.

### P3-8

## INTERNAL FRICTION, MODULUS AND RESISTANCE ASSOCIATED WITH TRANSFORMATION IN LANTHANUM

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Pure La transforms martensitically from FCC to dHCP with  $M_s=473K$  and  $A_s=573K$ . In order to study the transformation, combined measurements of internal friction ( $Q^{-1}$ ), modulus (resonance frequency ( $f$ )) and resistance ( $R$ ) were performed in a temperature region of 300K–700K with an inverted torsion pendulum. The main results are as follows.

1.  $Q^{-1}$ ,  $f$  and  $R$  vs.  $T$  curves obtained under the same measurement conditions depend on sample state and the number of thermal cycle. The transformation amount is much less in the cold-worked sample than in the annealed one. Even for the latter it is not possible yet to transform completely into dHCP phase at room temperature.
2.  $Q_m^{-1}$  vs.  $\dot{T}$  curve is similar to that of  $Q_m^{-1}$  vs.  $1/f$ . The relationship between  $Q_m^{-1}$  and  $\dot{T}/f$  is nonlinear and does not go through the original point.
3. In the transformation regions, both forward and reverse, constant  $\dot{T}$  measurements with holding at different temperatures show that isothermal transformations exist in La and austenitic (martensitic) stabilization can occur during temperature holding.
4. The variation of  $Q^{-1}$ ,  $f$  and  $R$  during holding or changing temperature again can be also seen in so-called nontransition region.  $R$  decreases when, during heating from 300K, temperature is kept even though it is far lower than  $A_s$ . A small thermal cycle in this nontransition region will result in a hysteresis loop of  $R$  and the higher the  $\dot{T}$ , the wider the loop is. But during cooling from 700K (FCC phase) a small thermal cycle in a temperature region nearby and higher than  $M_s$  won't result in the hysteresis loop. The phenomenon occurred in the dual-phase sample might be associated with instability of either the phases or the transformation induced defects.

### P3-9

## INFLUENCE OF HEAT TREATMENTS ON DAMPING BEHAVIOUR OF MARTENSITIC TRANSFORMATION IN Cu-Al-Ni-Mn-Ti ALLOY

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In this paper, the influence of heat treatments on damping behaviour of martensitic transformation is examined by measuring internal friction  $Q^{-1}$ , elastic modulus  $E/E_0$ , electric resistance  $R/R_0$  and zero point shift  $\theta$  vs. temperature  $T$  synchronously in the temperature range  $0^\circ\text{C}$  to  $160^\circ\text{C}$ . The sheet alloy of Cu-13Al-5Ni-1Mn-1Ti was rolled at about  $800^\circ\text{C}$  in several stages, then cooled to room temperature. The sheet was cut into strips for use of simultaneous measurements mentioned above. The 7 specimens were aged for 0.5 hour at  $T_a = 560, 590, 620, 650, 700, 800$  and  $900^\circ\text{C}$  respectively, then quenched into water. It is found that (1) for the specimens aged at  $T_a < 560^\circ\text{C}$ , there is not internal friction peak in  $Q^{-1}-T$  curve and the curves of  $E/E_0-T$ ,  $R/R_0-T$  and  $\theta-T$  have no abnormal phenomena, the specimen has no memory either. The x-ray diffraction and metallograph observation show that the M transformation does not occur. (2) for the specimens aged at  $T_a > 560^\circ\text{C}$ , the case is just the contrary. It is noticeable that two new phases,  $\text{Al}_{60}\text{Mn}_{11}\text{Ni}_4$  and  $\text{Cu}_{0.035}\text{Ni}_{0.565}\text{Ti}_{0.040}$ , are found. The report has not been seeing up to now.

### P3-10

## THE ULTRASONIC INSTABILITY OF SMALL-ANGLE BOUNDARIES NEAR THE POINTS OF MARTENSITIC PHASE TRANSFORMATIONS

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It is known that the influence of crystal defects affects mechanical properties of solids and it can greatly increase in the vicinity of phase transition (PT) points. In its turn, the process of PT can be accompanied by the important changes of defect structures in solids.

We have considered the dislocation model of the small-angle boundary (SAB) and applied the theory of "coating", developed earlier [1] for martensitic PT in crystals with dislocations.

We showed that the existence of "coated" SAB becomes energetically disadvantageous in the temperature interval about 0.1-100 K for different types of PT. This instability can be realized under the action of ultrasonic waves, parameters of which (such as wavelength and amplitude) have been estimated.

The methods of experimental observations of these anomalies are discussed.

1. A.L.Korzhenevskii, D.A.Lisachenko - Ferroelectr.Lett.(1991), v.12(6), p.135-141.

**P3-11**  
**ANHARMONIC BEHAVIOUR OF CuZnAl ALLOYS**  
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Internal friction and Young's modulus have been measured as a function of temperature in a CuZnAl alloy at varying amplitudes of the resonant flexural vibrations. It has been found that anharmonicity is low in the parent  $\beta$  phase and only shows slight premonitory effects of the martensitic transition above  $M_s$ . The alloy system becomes very anharmonic in the transition region, where the amplitude dependence of the Young's modulus passes through a pronounced maximum. The source of such anharmonicity is believed to be related to the lattice metastability. Below  $M_f$  anharmonicity is high, approximately temperature independent and, likely, it is associated with hysteretic motions of martensite-martensite interfaces.

**P3-12**  
**INTERNAL FRICTION IN Al-Li BINARY ALLOYS**  
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The precipitation process in Al-Li binary alloys has been very extensively studied but is not yet fully resolved. Nevertheless, there exist very few works in this kind of alloys using anelastic techniques. After quenching, the solid solution  $\alpha$  decomposes in the metastable and coherent phase  $\delta'$  which nucleates homogeneously through the matrix. Subsequent aging produces the precipitation growth. Finally this phase transforms to the stable  $\delta$  phase at the grain boundaries.

In this work, internal friction and modulus measurements have been carried out as a function of temperature using an inverted torsion pendulum at 1Hz. Our results, show that the sequence of  $\delta'$  and  $\delta$  precipitation can be followed through the evolution of the spectra. In parallel, the microstructural state of the alloy has been studied by X-ray powder diffraction measurements and electron microscopy.

The precipitation of  $\delta'$  coherent phase produces the growth of the background as it has been proposed by the Schoeck's theory. Also, this precipitation produces a change in the dynamic modulus of the alloy associated with the  $\delta'$  hardening. Several studies seem to indicate that the first stage of  $\delta'$  precipitation can not be avoided by quenching. In this work we show that the quenching has to be carried out under some necessary conditions in order to avoid  $\delta'$  precipitation. Furthermore, two peaks appear in the spectrum. The low temperature peak (450 K), associated with a decrease in the dynamic modulus, has an activation energy of 1.2 eV. The origin of this relaxation, usually found in Al and Al alloys, is discussed in the paper. The high temperature peak (560 K) associated with an increase in the dynamic modulus could be associated with the process of  $\delta' \rightarrow \delta$  transformation.

### P3-13

## HIGH TEMPERATURE INTERNAL FRICTION IN 2024 ALUMINIUM ALLOY

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A 2024 aluminium alloy was studied by isothermal mechanical spectrometry between 270 K and 820 K. Samples first were quenched. Experiments were carried out after a long time at each measurement temperature for specimen stabilization. Under a such experimental procedure, precipitation peaks as described by various authors were absent. But between 550 K and 750 K, an internal friction effect superimposed to a low frequency background was observed. This effect is situated always at the same frequency and increases with measurement temperature.

Because this effect disappears for measurements carried out after annealing above 800 K, it can be associated with the  $\theta$  precipitates.

A reversible phase transformation mechanism at the matrix - precipitate interface is proposed to explain this thermally non-activated effect.

### P3-14

## INTERNAL FRICTION IN Fe-Mn-Cr-Si-Ni SHAPE MEMORY ALLOYS

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It has been observed for the last years that several Fe base alloys present a shape memory effect. However, these alloys have a behaviour that is absolutely different from that of the shape memory alloys in Cu base and Ti base. All this has caused a growing scientific and technologic interest for these alloys.

In this work we have carried out Internal Friction and G-modulus measurements on Fe-Mn-Cr-Si-Ni samples, using an inverted torsion pendulum at 1 Hz.

The internal friction peaks associated to the martensitic transformation are clearly observed in the obtained spectra. They show a broad thermal hysteresis (around 100°C) as a consequence of the non-thermoelasticity of these alloys. We have been able to determine the evolution of the hysteresis versus the cycling, as well as the influence of an overimposed static stress.

The analysis of the internal friction spectra allowed us to obtain a lineal relationship between the peaks' area and the heating or cooling rate during the direct and reverse transformations. We also found a linear relationship between the accumulated integral of the internal friction and the quantity of transformed phase. Both aspects are discussed and are in good agreement with Delorme's model.

On the other side, in the curves of G-modulus we measured the Néel temperature ( $T_N$ ) associated to the paramagnetic-antiferromagnetic transition shown by these alloys and which interfere strongly with the martensitic transformation. The obtained  $T_N$  values were verified by magnetic measurements carried out with a Faraday balance.

P3-15  
NONLINEAR INTERNAL FRICTION PEAK ASSOCIATED WITH PHASE PRECIPITATION IN Al-Li ALLOYS

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Aluminium-Lithium alloy is a new type of aeronautic material with high strength, elastic modulus and low density. Precipitation is an essential feature of such alloys and can strongly influence mechanical properties. Nevertheless, the studies on its dynamic behavior and its distribution in Al-Li alloys are highly required.

In the paper, low frequency internal friction study was made on the precipitation of alloying element and the behavior of precipitates in Al-Li-Zr-Ti alloys. An temperature internal friction peak (called  $\beta$ -peak) was firstly observed near 240°C. It exhibits remarkable anomalous amplitude-dependent effects and the softening of elastic modulus. Heat cycling can lead to the disappearance of the peak and the decrease of elastic modulus in the whole temperature range. Frequency of measurement applied considerable influence on the peak. It was suggested that  $\beta$ -peak is resulted from the stress-induced movement of phase boundary of precipitates ( $\text{Al}_3\text{Li}$ ).

P3-16  
ACOUSTIC ANOMALIES IN ORIENTATIONAL PHASE TRANSITIONS

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Effect, known as "frozen lattice", is of common physics nature since it appears as a result of the spontaneous disturbed symmetry (spontaneous deformation) in the interacting fields' system in magnetics. This effect becomes apparent in the vicinity of the orientational phase transitions (OPT). The basic differences in the dynamics of OPT in orthoferrites are established through acoustic investigations. The present phenomenological theory describes the spontaneous deformation in magnetics fundamental state as the homogeneous sample deformation along the direction of spontaneous magnetization. Experimental data, however, differ considerably from the corresponding theoretical estimates. We propose a quantitative analysis of such kind experiments on the basis of the microscopic approach to the problem. The last one is based on the ideas about the local soft optic and acoustic phonon (photon) modes condensation, i.e. the spontaneous deformation locality in fundamental state of magnetics. No results of measurements are used for numerical estimations. For example, if in the case of the low-temperature PT in  $\text{ErFeO}_3$  the calculated  $T_n=4,12\text{K}$ , then at a temperature  $T>T_n$  the acoustic impulse spreads with  $V_1=3,81\cdot 10^5\text{ cm/s}$  along the string consisting of the chain of atomic oxygen magnetic ions. The latter are exchange-connected through the interstitial diamagnetic 2-valent oxygen anion. The fluctuation range of the order parameter is  $\delta T_n=0,4\text{K}$ . In this range begins the formation of the second string (the chain of erbium ions) and its coupling with the first string through the 2-valent oxygen. It is accompanied by the short-range magnetic order appearance in the erbium subsystem and comparatively smooth sound velocity decrease to  $V_2=3,29\cdot 10^5\text{ cm/s}$ . Under  $T<T_n$  the long-range magnetic order forms itself in the erbium subsystem and the acoustic impulse velocity increases rapidly to  $V_3=3,89\cdot 10^5\text{ cm/s}$ . The maximum sound damping is  $\Gamma=119,49\text{ dB/cm}$  and corresponds to  $T=T_n$ . Experimentally measured parameters are:  $T_n=(4,1\pm 0,03)\text{K}$ ;  $\Gamma=110\text{ dB/cm}$ ;  $V_1=(3,81\pm 0,05)\cdot 10^5\text{ cm/s}$ ;  $V_2=(3,2\pm 0,02)\cdot 10^5\text{ cm/s}$ ;  $V_3=(3,89\pm 0,04)\cdot 10^5\text{ cm/s}$ . Obviously we have obtained the satisfactory quantitative agreement between theory and experiment. This significant fact stimulates the development of the alternative theoretical method.

P3-17  
INTERNAL FRICTION AND TI-RICH PRECIPITATION IN NiTi ALLOY

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Internal friction (IF) and modulus ( $\propto f^2$ ), electric resistance and zero position shift were measured in Ti-rich NiTi alloy before and after aging at 500°C for over 1h. An anomaly of IF and corresponding to a variation of modulus, electric resistance and zero position shift below  $M_s$  were observed in aged sample. A further experiment indicated that the anomaly of IF had the characteristic of first order phase transition i.e peak high of IF is proportion to the temperature rate and inverse proportion to the measuring frequency. SME and x-ray diffraction technique were used to clarify the observed phenomena. Grain shape precipitates which have Ti-rich composition were found by SME in aged sample. It is also confirmed by x-ray diffraction, large enhancement of diffraction lines intensity for NiTi<sub>2</sub> phase was observed in this sample. As we know, the appearance of Ti-rich precipitate phase reduces Ti content in area near precipitate phase. A decrease of martensitic transition point in this area was found because it has more Ni content. The observed anomalous below  $M_s$  are attributed to the martensitic transition in the area with lower  $M_s$  point in aged sample.

**P4-1**  
**THE INFLUENCE OF HYDROGEN AND DISLOCATIONS ON THE INTERNAL FRICTION IN YTTRIUM**

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The anelastic relaxation of polycrystalline hexagonal Y samples was studied (i) after a doping with H or D ( $\alpha$ -phase  $YH_x$  or  $YD_x$  with  $x \leq 0.17$ ) and (ii) after a plastic deformation (up to 20 %). The study was carried out in the temperature range between 5 and 330 K with the help of the vibrating reed technique (frequencies  $f$  between 100 and 1100 Hz). The H and D doped samples exhibit a single relaxation peak located, e.g. for  $f = 500$  Hz, at 272 and 279 K, respectively. For both H and D, the relaxation shows an Arrhenius behavior with an activation energy  $E = (0.60 \pm 0.05)$  eV. The results are in agreement with previous studies [1,2]. The plastic deformation causes a relaxation peak with three striking properties in comparison to that one found after H or D doping. The relaxation peak following plastic deformation is considerably larger, its position is at only slightly lower temperatures (up to 19 K lower for  $f = 500$  Hz) and its temperature dependence exhibits an Arrhenius relation with an identical activation energy. The microscopic mechanisms which may be responsible for these striking properties are discussed.

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**P4-2**  
**GORSKY EFFECT IN Nb-46%Ti-H ALLOYS**  
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The Gorsky Effect is investigated by internal friction measurements in the niobium-titanium-hydrogen system with 46% wt. titanium. The internal friction measurements were made in a torsion pendulum with frequency at 3.8 Hz in a temperature range from 80 to 700 K. The diffusion coefficient,  $D$ , obeys the Arrhenius' law in the temperature range from 300 to 700 K, with  $D_0 = 7.43 \times 10^{-4}$  cm<sup>2</sup>/s and activation energy  $E = (0.102 \pm 0.003)$  eV. At low temperatures, it was found a deviation from Arrhenius' law, with  $D_0 = 0.37 \times 10^{-4}$  cm<sup>2</sup>/s and  $E = (0.063 \pm 0.002)$  eV. (Financial support: CNPq and FUNDUNESP).

P4-3

ANELASTIC RELAXATION DUE O-H PAIRS IN THE Nb-Zr ALLOYS

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In the last thirty years several studies have been made to understand the relaxations mechanisms of the hydrogen atoms presented in transition metals and their alloys. In this work, we observed the stress induced ordering of hydrogen atoms around the oxygen atoms near the niobium matrix atoms. We studied this relaxation process by measuring the attenuation of the longitudinal ultrasonic waves. These measurements were made in Nb-1.0% Zr polycrystalline alloys on 10 and 30 MHz, pure and doped with 0.5 and 5.0% at. of hydrogen. The results showed a thermally activated relaxation structure around 202 and 235 K for 10 and 30 MHz, respectively. This relaxation structure increases with the increasing of hydrogen concentration. (Financial support: CNPq and FUNDUNESP).

P4-4

INTERNAL FRICTION OF THE HYDROGEN CHARGED AUSTENITIC STEELS DUE TO THE HYDROGEN DESORPTION

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Internal friction peak of nonrelaxation nature was found in hydrogen charged austenitic steels at low frequencies (0.1-10 Hz) at temperatures 270-350 K. Peak amplitude and its temperature position essentially depends on the heating rate and frequency. Peak mechanism is proposed according to which losses of mechanical energy of torsional oscillations are related with the hydrogen diffusion flows during its desorption from the specimen. Expressions for the estimations of the hydrogen diffusive mobility in metal were obtained from the parameters of the peak in question. Proposed model was extended on the other cases of inhomogeneous and nonstationary change of nonrelaxed shear modulus.

**P4-5**

**HYDROGEN ANELASTIC PROCESSES IN PALLADIUM-SILVER ALLOYS: NEW DATA AND INTERPRETATION**

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Anelastic investigations have been carried out over an extended range of silver contents in PdAg alloys containing H. Evidence of smoothly systematic variations of the H(1) and H(2) peaks have been obtained and accounted for in terms of gradual alterations of occupancy of different types of interstitial sites. For peak H(2) mechanisms alternative to that of stress induced changes in the short-range order of H (Zener effect) are considered and discussed.

**P4-6**

**RELAXATION PARAMETERS AND ANNEALING PROCESS OF H-INTERNAL FRICTION PEAK IN AN AUSTENITIC STAINLESS STEEL**

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H-internal friction peak in an austenitic stainless steel, type 2179, has been studied with an inverted torsion pendulum. Residual H in the original sample, annealed at 1323K and then quenched into water, is 3.9 wtppm. H content increases to 72 wtppm after high pressure gas thermo-charging. An internal friction peak of  $3.7 \times 10^{-4}$  in height occurs at 240K (1 Hz) in the charged sample. Whereas in the original sample there is only a hillock of  $\sim 0.3 \times 10^{-4}$  in height around the same temperature. The relaxation parameters of the H-peak are:  $H = 0.563\text{eV}$ ,  $\tau_0 = 3.4 \times 10^{-13}\text{s}$ . In comparison with the results of H permeability experiment performed on the steel it can be seen that the activation enthalpy is higher and the diffusion coefficient at the peak temperature, converted by  $D = a^2 / 6\tau$ , is lower for the internal friction experiment. The difference in both enthalpy and diffusion coefficient exceeds the generally recognized error coming from the two kinds of measurement methods. This contradiction can be resolved on the basis of defect-pair reorientation model for the H-peak. A new conversion method has been proposed. The result is well.

In-situ annealing gives rise to a decrease in H-peak height. By means of a two-dimension diffusion model and a stress expression in a rectangle-section sample an expression of the apparent internal friction in the sample, as a function of annealing process has been deduced for H-H pair and H-S pair mechanisms respectively. The experiment data agree very well with the theoretical curve calculated on the H-S pair. Furthermore a comparison of the ratio of peak height to H content for the original sample with for the charged sample seems also to support the H-S pair mechanism.

P4-7  
HYDROGEN INTERNAL FRICTION PEAK AND THERMAL DESORPTION SPECTRUM IN  
AMORPHOUS  $\text{Cu}_{60}\text{Zr}_{40}$

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Hydrogen is a useful probe to provide an insight into the atomic structures in amorphous alloys, especially in early transition metal-late transition metal amorphous alloys (ETM/LTM a-alloy). For ETM/LTM a-alloys, both Harrison et al. and Kirchheim et al. found that hydrogen occupies the tetrahedral sites under the nearest neighbor blocking, and we found that only hydrogen atoms sitting in the sites with the site energy  $G=\mu$  can contribute to the hydrogen diffusion in the materials, where  $\mu$  is the chemical potential of hydrogen in the materials. These facts allow us the investigation relevant to the amorphous structure in ETM/LTM a-alloys. In the present paper, we investigated the hydrogen internal friction peak (HIFP) and thermal desorption spectrum (HTDS) in  $\text{a-Cu}_{60}\text{Zr}_{40}$ .

$\text{a-Cu}_{60}\text{Zr}_{40}$  was prepared by the melt-spinning method. Hydrogen charging was made electrolytically. HIFP was measured by the vibrating-reed technique, and HTDS by the vacuum extraction method. In  $\text{a-Cu}_{60}\text{Zr}_{40}$ , HIFP is observed as a broad peak extending from 5 to 350K showing a maximum near room temperature. The total relaxation strength,  $S_t$ , of HIFP shows the repetitions of an increase followed by a decrease with increasing the hydrogen concentration,  $C_H$ . The observed  $C_H$  dependence of  $S_t$  can well be explained by the model mentioned above; the Gaussian distribution  $N_1(G)$  for 4Zr sites for  $C_H \leq 6\text{at.}\%$ , the two Gaussian distributions  $N_2(G)$  and  $N_3(G)$  for 3Zr+1Cu sites for  $6\text{at.}\% \leq C_H \leq 30\text{at.}\%$ , and the Gaussian distribution  $N_3(G)$  for 2Zr+2Cu sites for  $C_H \geq 30\text{at.}\%$ , respectively. These features for HIFP are very similar to those reported for  $\text{a-Cu}_{50}\text{Zr}_{50}$  and  $\text{a-Cu}_{50}\text{Ti}_{50}$  after taking into account the change in the chemical composition except that HIFP in  $\text{a-Cu}_{60}\text{Zr}_{40}$  is relatively narrower compared with that in  $\text{a-Cu}_{50}\text{Zr}_{50}$  and  $\text{a-Cu}_{50}\text{Ti}_{50}$ . Meanwhile the HTDS data suggested that the heat of solution of hydrogen into the 4Zr sites in the as-quenched  $\text{a-Cu}_{60}\text{Zr}_{40}$  is about  $-50\text{kJ/mol.H}$ , which is much larger than about  $-34\text{kJ/mol.H}$  for the 4Zr sites in  $\text{a-Cu}_{50}\text{Zr}_{50}$ . These facts suggest that even in the as-quenched state, the free volume in the a-structure is presumably smaller in  $\text{a-Cu}_{60}\text{Zr}_{40}$  than in  $\text{a-Cu}_{50}\text{Zr}_{50}$ .

P4-8  
MECHANICAL RELAXATION OF HYDROGEN-SOLUTE COMPLEXES IN NICKEL

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Atomistic configuration of Ti-H complexes in Ni have been studied by measuring anisotropy of relaxation strength of the internal friction due to the reorientation of the complexes. Low-frequency internal friction measurements have been made on Ni-1 at.% Ti single crystal specimens using a torsion pendulum apparatus. The orientation dependence of the peak height indicates that the defect symmetry is  $\langle 100 \rangle$  tetragonal, which is consistent with the configuration so far assumed: hydrogen is located at the nearest neighbour octahedral site of the substitutional solute atom. Application of external magnetic field has been found to suppress the internal friction peak. This suggests the peak involves some magnetic origin.

A similar internal friction peak has also been observed in Ni-C-H alloys, which is attributed to the reorientation of C-H pairs; results of the measurements on these alloys will also be reported.

**P4-9**

**MEASUREMENTS OF HYDROGEN SOLUBILITY DURING ISOTHERMAL CHARGING IN Zr ALLOYS USING AN INTERNAL FRICTION TECHNIQUE**

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A linear relationship between Young's Modulus and the hydrogen concentration in solid solution of Zr alloys at a given temperature has been reconfirmed by the present experiments. Therefore, the increment of hydrogen concentration in solution during ingress of hydrogen from a gas phase into a Zr alloy sample can be monitored continuously by measuring Young's modulus as a function of time at constant temperature. The effects of supercharging and the process of supersaturation of hydrogen above the dissolution terminal solid solubility (TSS) of hydrogen in Zr alloys was observed by the piezoelectric ultrasonic composite oscillator technique set to a frequency of 40kHz. The hydrogen concentration at the beginning of saturation in the change of the Young's modulus with time during charging was identified as the TSS. This TSS was observed to be close to the precipitation TSS determined in the same Zr alloy by measurements on samples containing fixed amounts of hydrogen. The effect of thermal cycles on the hydrogen supercharging process was investigated. The connection between the dissolution and precipitation TSS and the hydrogen supercharging process is discussed.

**P4-10**

**SOME ASPECTS OF THE HYDROGEN REORIENTATION RELAXATION IN AMORPHOUS ALLOYS**

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Although the main features of hydrogen absorption in amorphous alloys appear well understood since several years, a really satisfactory model of the H reorientation relaxation is still missing. It is well known that the broad distribution of site energies implies an occupation according to Fermi-Dirac statistics. However, this statistics must not be used for the relaxation process (thermally activated hopping between neighbouring interstitial sites) unless taking into account that for a given distribution of H atoms, there are more sites available for hopping of these atoms than for adding new H atoms. This follows directly from the local H-H repulsion that prohibits simultaneous occupation of any two directly neighbouring sites. Consequently, all those models which assume a relaxational contribution only from H atoms with site energies near the Fermi level are basically inconsistent with H-H repulsion. Other models, however, have failed to explain the decrease of relaxation strength observed in several glasses at higher H concentrations.

Considering the elementary relaxational contribution from any "new" H atom added to a given distribution, there are a positive and a negative part: the H atom acts as a new relaxation centre, but also reduces the hopping possibilities of previously present atoms if there is a mutual overlap of the empty nearest-neighbour shells of sites. With increasing H concentration, three characteristic ranges can (at least) be distinguished: (1) the negative part is negligible, (2) it is still small but causes reduction of high-activation-energy processes, (3) it becomes comparable to or larger than the positive part. These three ranges are indeed observed experimentally, e.g. in amorphous  $\text{Co}_{33}\text{Zr}_{67}$  when the H concentration is varied over three orders of magnitude up to 1 H/metal atom. Differences between alloys may be explained by a shift of these ranges along the H concentration scale; possible reasons for such a shift are discussed.

**P4-11**  
**HYDROGEN-INDUCED INTERNAL FRICTION IN  $\text{NiZr}_2$**   
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The H-induced internal friction in the C16 phase  $\text{NiZr}_2$  was measured using the vibrating-reed technique combined with in-situ hydrogen charging from the gas phase, on samples crystallized from amorphous ribbons with compositions slightly varying around the stoichiometric value. At H concentrations of about 0.3-1 at%, two damping peaks at temperatures near 160 K and 250 K (for a frequency of 300 Hz) are observed in most of the samples, the former being distinctly broader than the latter. At higher H concentrations, a third peak enters into the temperature range investigated ( $T > 90\text{K}$ ) from low temperatures. Both this low-temperature peak and that one near 250 K grow faster with increasing H concentration than the broad 160 K peak in between, so that the latter is eventually masked.

According to a recently published model of the H reorientation relaxation in C16 compounds (tetragonal  $\text{CuAl}_2$ -type structure; see H.-R. Sinning, Phys. Rev. B 46(1992) 5989), the peaks near 160 K and 250 K are mainly attributed to the two types of reorientation jumps (called "direct" and "indirect") between the Zr-surrounded tetrahedral sites in the 16 l positions of the C16 lattice. However, the observed width of the peaks suggests an important influence of H-H interactions not considered in the model, which appears stronger for the direct than for the indirect jump.

In addition, a "reaction mode" of relaxation by exchanging H atoms between the 4b and 16 l sites may also be possible in the C16 structure, probably at relatively low temperatures. The question is discussed if this could be an explanation of the third peak discovered.

**P4-12**  
**INTERNAL FRICTION DUE TO HYDROGEN MOTION IN Pd-BASED DILUTE ALLOYS**  
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Low frequency internal friction ( $\sim 1\text{ Hz}$ ) of Pd-M-H alloys ( $M = \text{Nb, Cu, Au, Ag}$ ) has been studied over a temperature range of 5 to 300 K. For all the alloys with 1 at.%M and about 1 at.%H, two internal friction peaks are observed below 150 K. A lower temperature peak around 75 K is considered to be due to the short range ordering of hydrogen atoms in hydrides (the Zener effect). A higher temperature peak around 100 K which increases by plastic deformation is identified as the hydrogen cold work peak. Dislocations generated during hydride precipitation are responsible for this peak. The behaviour of these peaks is almost the same as that observed in a Pd-H alloy. The solute concentration dependence of the internal friction has also been studied for Pd-Cu alloys (1 - 20 at.%Cu). In these alloys the Zener peak shifts toward higher temperatures and its width increases with increasing Cu concentration.

A hydrogen-solute atom interaction peak has not been observed for all these alloys. This is in contrast with Ni-based fcc alloys which show the interaction peak[1]. Thus it is concluded that the hydrogen-solute atom interaction is not strongly attractive in these Pd-based alloys.

[1] O. Yoshinari, K. Sanpei and K. Tanaka: *Acta Metall. Mater.*, **39** (1991) 2657.

**P4-13**  
**ON ORIGINS OF ANELASTIC EFFECTS IN HYDROGEN CHARGED NICKEL AND Ni-BASE ALLOYS**

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The results of so far internal friction and ultrasonic attenuation investigations on hydrogen charged nickel, some nickel alloys and austenitic stainless steels are presented. Neither in hydrogen charged nickel nor in  $\text{Ni}_3\text{Al}$  alloy any internal friction peaks have been found in low frequency tests. On the contrary, in stainless steels, Fe-Ni and Cr-Ni alloys, a number of relaxation peaks, between one and five, has been discovered at low and medium frequency by various investigators; heights of the peaks are shown to relate on hydrogen concentration and on susceptibility of alloy to some phase transformations. The ultrasonic wave study on hydrogen charged nickel single crystal has disclosed modification of attenuation which seems to originate from a Snoek-like relaxation mechanism. The hydrogen charging has influenced on magnetic and non-magnetic background, at both low and high frequency, in a manner dependent on hydrogen charging conditions.

An absence of any low frequency relaxation peak in Ni and  $\text{Ni}_3\text{Al}$ , and complex relations of peak height on hydrogen concentration and on chemical composition in steels suggest that the origin of low frequency peak in steels is likely the relaxation of Me-H complexes in one or two solid phases rather than of H-H atom pairs. The possible interaction of hydrogen and dislocations as the source of the high temperature satellite peak in steels is also discussed. The increase in attenuation observed in hydrogen charged high purity nickel may be associated with an appearance of relaxation of the Ni-H atom pairs. The disappearance of Bordoni relaxation and decrease in dislocation damping are suggested to be caused by hydrogen pinning of dislocations. The increase in magnetic hysteretic internal friction and magnetic part of ultrasonic attenuation may relate to the effect of hydrogen on the movement of magnetic domains.

**P4-14**  
**REORIENTATION OF H TRAPPED BY Zr IN Nb SINGLE CRYSTALS**

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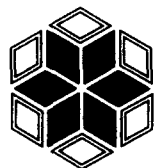
The anelastic relaxation due to the reorientation of the Zr-H pair has been measured in two Nb single crystals at low H and Zr contents. The two crystals were oriented with the longer dimension along the [100] and [111] directions, and were excited on their extensional, torsional and flexural modes. The observed dissipation between 50 and 200 K is composed of at least two peaks. One of them is not observed under [100] torsional and [111] extensional vibrations, and this is interpreted as due to the redistribution of H among four-site tunnel systems over the faces of the cube containing the Zr atom. The other peak is predominant and is always observed, implying the occupation of additional sites.

# **Cryophysics** **l'altro modo di** **scoprire**

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**P5-1**  
**THE EFFECT OF NEUTRON AND GAMMA IRRADIATION ON YBCO AND BPbSCCO HIGH  $T_c$ -SUPERCONDUCTOR**

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The effect of neutron irradiation in YBCO high  $T_c$ -superconductor ( $T_c = 93$  K) and the effect of  $\gamma$ -irradiation in BPbSCCO high  $T_c$ -superconductor ( $T_c = 87$  K) has been studied. Each sample was given successive irradiation for 5, 10, 15, 20 and 30 hours. The mechanical loss spectra have been recorded for temperature from liquid nitrogen to room temperature. In the former sample (YBCO), the height of the five observed relaxation peaks increase as the neutron dose was increased. The peak heights increase nearly by a factor of two (when comparing the neutron irradiation for 30 hours with the non-irradiated sample). In the later sample (BPbSCCO) the peak heights increase by a factor of 1.5 (for 30 hours  $\gamma$ -irradiation). The peak temperatures in both high  $T_c$ -superconductor show nearly no change as the neutron or gamma irradiation increases. In the YBCO sample the peaks always occur at 113, 123, 147 and 243 K, while in BPbSCCO the peaks always occur at 87, 112, 128, 170 and 230 K, respectively, when measurement is carried out at 10 MHz using the pulse echo technique (single ended).

**P5-2**  
**STOICHIOMETRY DEPENDENCE OF OXYGEN ANELASTIC RELAXATION IN  $YBa_2Cu_3O_{7-x}$  AND THE ORTHORHOMBIC TO TETRAGONAL PHASE TRANSITION**

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Measurements of the elastic energy dissipation coefficient and of the dynamic elasticity moduli, have been performed on polycrystalline YBCO specimens by the vibrating reed technique and torsional pendulum in the  $10$ - $10^4$  Hz range. Tests have been conducted in isothermal and isochronal conditions, in different oxygen pressure and for different and carefully controlled stoichiometries.

From the isothermal modulus measurements, details have been obtained on the orthorhombic to tetragonal phase transition kinetics. Two stages have been identified contributing to the transformation. Long times modulus isotherms of exponential type give an activation energy  $0.9 \pm 0.1$  eV.

The isochronal elastic energy dissipation measurements show two peaks P01, P02 whose nature is intrinsically relaxational. A careful investigation of peaks parameters: activation energies, relaxation strength, peak shape and peak temperature, unequivocally indicate that they may be attributed respectively to short range oxygen dynamics in the well oxygenated twinned superconducting phase and in the superconducting substoichiometric ones. The evolution of these peaks with oxygen in-out diffusion was carefully investigated. Phenomenological models for the structural transformations and the dynamics of oxygen in different stoichiometric conditions are discussed and compared with those derived by other authors.

### P5-3

## STUDY OF OXYGEN BEHAVIORS IN Bi-SYSTEM SUPERCONDUCTORS BY INTERNAL FRICTION MEASUREMENTS

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Low frequency internal friction (IF) measurements have been carried out for the 2223 phase ( $\text{Bi}_{1-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+x}$ ) and the 2212 phase ( $\text{Bi}_2\text{Sr}_{2-x}\text{Ca}_{1+x}\text{Cu}_2\text{O}_8$ ) superconducting specimens subjected to in situ vacuum (20Pa) thermal cycling in different temperature regions. It was found that for the as-prepared 2223 specimen there exists a broad and asymmetrical IF peak located at 130°C ( $f = 5.8$  Hz), and during successive vacuum cycling the peak shifts gradually towards higher temperature and shows complicated changes in height, decreasing in the early stage and increasing afterwards. By means of computer fitting, all of the IF peaks can be well decomposed into two sub-peaks,  $P_i$  and  $P_v$ , located at 127°C and 170°C with relaxation activation energies of 0.66 eV and 1.10 eV respectively. It is interesting that with decreasing oxygen content in the 2223 phase, the  $P_i$  peak drops while the  $P_v$  peak rises in height. From this fact, we concluded that there exist two kinds of oxygen defects in the double  $\text{Bi}_2\text{-O}_2$  layers: the isolated interstitial oxygen atoms and the oxygen vacancies in the one-dimensional excess oxygen chains. On the other hand, a result from Hall measurement exhibited that with oxygen content, the changes of the charge carrier concentration and the height of the  $P_i$  peak are very similar. So that, we believed that the isolated interstitial oxygen would play a crucial role in determining superconductivity in the 2223 phase superconductor. The IF behavior for the 2212 phase compound was also studied, and it is similar to that for the 2223 phase, but the height of the IF peaks of the 2212 phase are much higher than that of the 2223 phase, indicating that more excess oxygen is inserted in the double  $\text{Bi}_2\text{-O}_2$  layers for the 2212 phase superconductor.

### P5-4

## ULTRASOUND ATTENUATION AND VELOCITY IN $\text{Bi}_2\text{Sr}_{2-x}\text{Ca}_{1+x}\text{Cu}_2\text{O}_8$

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We have measured ultrasound attenuation and velocities in  $\text{Bi}_2\text{Sr}_{2-x}\text{Ca}_{1+x}\text{Cu}_2\text{O}_8$  samples with  $x=0, .25, .50, .75, 1$ . The attenuation has a maximum near to 240 K in all samples. The velocities increase upon cooling, as expected. However, this increase depends on  $x$  for the longitudinal velocity and it is almost independent on  $x$  for the shear velocity.

**P5-5**  
**OXYGEN MOBILITY IN  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  - I - EXPERIMENTAL RESULTS**

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The aim of this experimental study is to characterize oxygen mobility in CuO confined planes in the YBaCuO high  $T_c$  superconductor. The large scattering of oxygen relaxation parameters given by different authors indicates that oxygen stoichiometry, correlated to the degree of anisotropy, is the most important parameter influencing the apparent relaxation characteristics. Our intention is to describe oxygen relaxation as a function of its concentration by means of very low frequencies mechanical spectroscopy technique, allowing measurements in a temperature range where this concentration stays stable.

For oxygen rich specimen, corresponding to orthorhombic-I phase, we observe a single relaxation peak. The measured activation energy  $E_I$  varies between 0.8 and 1.3 eV and is a complex function of oxygen concentration. For less rich oxygen contents, a second relaxation peak is observed, coexisting with the above mentioned one. Its energy  $E_{II}$  is about 1.10 eV, independent of the concentration. However chemical and X-Ray analysis and phase diagrams prove that this second relaxation is associated with orthorhombic-II phase existence.

A second paper describes a theoretical analysis proposed to explain mainly the variations of relaxation characteristics in an anisotropic and concentrated medium and the possibility to observe both isotropic and anisotropic relaxation types in an anisotropic phase.

**P5-6**  
**OXYGEN MOBILITY IN  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  - II - THEORETICAL ANALYSIS**

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In this paper we propose to develop a microscopic model describing the relaxation of concentrated interstitials in an anisotropic medium ; it is applied to experimental results of oxygen mobility in YBaCuO displayed in another paper in this session. If different authors have taken into account the difference of energy sites and atoms populations characterizing CuO planes along a and b perpendicular axes, we introduce new conditions :

- Boltzman's law is not verified. Neighbouring sites probability of occupation introduces new jump frequencies and thermal equilibrium conditions.

- jump probabilities of interstitials of the same nature are correlated. This correlation do not act on relaxation parameters in isotropic media, but considerably changes the relaxation time in anisotropic media towards higher temperatures.

The model shows that the relaxation time do not follow an Arrhenius law, but apparent activation energies can be deduced in experimental window, and are in good agreement with experimental values obtained for different oxygen concentrations.

Thermal equilibrium is more complex to describe in orthorhombic II phase. However, we assume that two independent relaxations exist in this phase. The first is a Snoeck type relaxation between equivalent sites corresponding to relaxation in tetragonal phase. The second is associated to jumps between sites of different energy levels as described in orthorhombic I phase.

# P5-7

## ELASTIC MODULI OF SOME HTS SINGLE CRYSTALS

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We report the ultrasonic measurements of the sound velocities in the  $\text{La}(2-x)\text{Sr}(x)\text{CuO}_4$  and  $\text{Nd}(2-x)\text{Ce}(x)\text{CuO}_4$  single crystals at frequencies of  $\approx 50\text{MHz}$ . We used a novel version of the phase method elaborated for measurements of absolute value of the sound velocity in the strongly attenuating highly scattering materials (twinned during the phase transformations) and obtained the complete sets of the velocities within the accuracy not worse than 1% at two temperatures: 77K and 295K for two dopant concentrations:  $x=0, 0.13$  in both compounds. That enables us to derive the elastic moduli tensors and to calculate by numerical averaging the Debye characteristic temperatures of these crystals. In LSCO the temperature of orthorhombic phase transition determined acoustically agrees with other experimental measurements, e.g. for  $x=0.13$   $T=150\text{K}$ . An interesting result is that some elastic moduli decrease with  $x$ , particularly, a modulus  $(c_{11}-c_{12})/2$  decreases almost by two times at 77K: from  $4.6 \cdot 10^{12} \text{erg/cm}^2$  for  $x=0$  to  $2.53 \cdot 10^{12} \text{erg/cm}^2$  for  $x = 0.13$ . This softening reflects in the Debye temperature drop from 441K to 397K, respectively. Temperature dependencies of sound velocities in LSCO reveal another anomaly: marked softening of the  $c_{11}$  and  $(c_{11}-c_{12})/2$  moduli that is accompanied by a peculiarity in magnetic susceptibility along the  $\text{CuO}$  layers. The characteristic temperature of this anomaly is  $x$ -dependent (20K for  $x=0$  to 6K for  $x=0.125$ ). Its origin is connected most likely with potential lattice instability with respect to low-temperature phase transition of the type existing in the  $\text{LaBaCuO}$  structure.

# P5-8

## LATTICE THERMAL CONDUCTIVITY OF YBCO-SUPERCONDUCTORS

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The lattice thermal conductivity of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  compounds, have been theoretically investigated, by considering the scattering strengths due to boundary, point-defect, 3-phonon and the electron-phonon scattering. The role of interference terms between the point-defect and 3-phonon scattering, in the phonon relaxation rates, has been found important, near the low temperature ( $T < T_c$ ) conductivity maximum. The abrupt change of slope in  $K$  vs  $T$  curve near  $T_c$  and the presence of a weak maximum around 160K, has been explained by considering the relative strengths of various scattering terms. A good agreement between theory and experimental measurements has been achieved. The present analysis differs from those using Callaway's phenomenological model, with the view that we do not explicitly assume the validity of the inverse summation rule in the conductivity calculations, as done by earlier workers.

P5-9

LOW TEMPERATURE MECHANICAL ENERGY DISSIPATION PHENOMENA IN  
PRASEODYMIUM-DOPED YTTRIUM SUPERCONDUCTORS

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Studies of the anelastic properties of  $Y_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$  ceramics ( $0 < x < 1$ ) were performed. The internal friction and Young's modulus measurements were carried out by the vibrating reed technique in the temperature range from 20 to 170 K. The measurement frequency was in the range of 90 - 760 Hz.

The aim of this work was to investigate the influence of praseodymium contents on the anelastic properties of 1-2-3 material.

The main phenomena which are caused by doping with praseodymium can be seen in the low temperature part of the internal friction spectra (20 - 40 K).

The maximum at temperature about 40 K, known as maximum 1 in the spectra of yttrium 1-2-3 material, gradually decreases with the increase of praseodymium contents (for  $x < 0.6$ ). Finally, it disappears in the  $Y_{0.5}Pr_{0.5}Ba_2Cu_3O_{7-\delta}$  sample. Relaxation processes located in the conducting planes, possibly electronic phenomena, have been considered as an origin of maximum 1.

The samples which contained more than 70% of praseodymium ( $x > 0.7$ ) showed other two low temperature maxima observed at temperature 25 - 40 K. In the spectra of  $Pr_1Ba_2Cu_3O_{7-\delta}$  they are large and partially overlap.

The common feature of all the  $Y_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$  samples regardless of the dopant contents is the presence of internal friction maxima observed in our frequency range at temperature 80 K and 100 K (maxima 3 and 4 in the spectra of 1-2-3 material). Neither their temperature nor height was significantly affected by the presence of praseodymium.

P5-10

INTERNAL FRICTION AND ELASTIC TWIN DYNAMICS IN HIGH- $T_c$  SUPERCONDUCTORS

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High- $T_c$  superconducting Y-Ba-Cu-O ceramics undergo a structural ortho-tetra phase transition ( $P4/mmm - Pmmm$ ) near 920 K which is accompanied by the appearance of spontaneous shear strain and the ferroelastic twins originating in the (110) plane below 920 K. Nowadays there is no doubt that twin structure rearrangement greatly influences on mechanical properties of twinned samples. But there is no common point of view about a contribution of twin wall dynamics in the low-frequency internal friction of high- $T_c$  superconductors. The internal friction (IF) and shear elastic modulus measurements of Y-Ba-Cu-O samples were made by an inverted torsion pendulum at a frequency 1 - 20 Hz in a wide temperature range 50 - 950 K. A few pronounced IF peaks and corresponding shear modulus changes were observed near 70, 90, 240, 370, 550 and 910 K. The experimental results allow to conclude the influence of twin boundary content in a sample on the strength of relaxation processes near 90, 240, 550 and 910 K. The features of IF anomaly at 910 K show that the mechanical losses in the vicinity of first order ferroelastic transition are due to the movement of domain and phase boundaries through the stoppers. IF peak near 550 K is of the Debye type. The relaxation is supposed to be due to the process of interaction between the elastic twin boundaries and point defects (oxygen vacancies). It was found out that 240 K-peak noticeable increases with a temperature rate increasing, besides it is accompanied by a great shear modulus hysteresis. The results do not contradict the idea of martensite-like transition presence in the peak region. The IF peak near 90 K supposes to be due to the structural instability of the crystal lattice related to the twin dynamics.

**P5-11**  
**ULTRASONIC BEHAVIOR OF  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7-y}$  HIGH  $T_c$  SUPERCONDUCTOR**  
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The behavior of ultrasonic velocity and attenuation on  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7-y}$  high  $T_c$  Superconductor has been studied with Pulse-echo method from 15K to 300K at 5MHz and 10MHz. Ultrasonic anomalies have been observed near  $T_c$ , 170K and 240K. There are two attenuation peaks at 170K and 240K. The big peak at 240K is a phase transition one coming from anti-symmetric distortion of Oxygen atoms on Cu-O plane. This peak is not related to superconducting transition. The small peak at 170K maybe from Martensitic phase transition and motion of twins. There are also velocity anomalies at 170K and 240K. Near  $T_c$ , phonon soften has been found, and attenuation decreased, it is similar to the behavior in traditional superconductors.

**P5-12**  
**ELASTIC PROPERTIES OF FULLERENES**

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We report on ultrasonic measurements of both sound velocity and attenuation on samples of different  $\text{C}_{60}/\text{C}_{70}$  composition. Although the well-known phase transitions at about 160 K and 260 K are clearly seen in our experiments, concerning the absolute values of the sound velocity our results differ markedly from those found by means of low frequency vibrating reed techniques on pure  $\text{C}_{60}$ . The possible consequences for both the bonding and the superconducting pairing mechanism will be discussed.

**P5-13**  
**EFFECTS OF THE MAGNETIC FIELD ON THE INTERNAL FRICTION OF  $\text{YBa}_2\text{Cu}_3\text{O}_{6.9}$ -THIN FILMS**

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We present internal friction (IF) measurements of epitaxial  $\text{YBa}_2\text{Cu}_3\text{O}_{6.9}$  - thin films on  $\text{LaAlO}_3$  and  $\text{NdGaO}_3$  substrates in magnetic fields up to 3T. The film thickness was varied between 50nm and 600nm. Patterned films and films with an isolating  $\text{SrTiO}_3$  interlayer were also investigated in order to study effects related to the sample geometry.

For fields perpendicular to the c-axis of the films we find a sharp maximum in the temperature dependence of the IF slightly below  $T_c$ . For a small tilt of the samples with respect to the magnetic field the maximum broadens and shifts to lower temperatures. The occurrence of these maxima can be explained by means of thermally assisted flux flow (TAFF). The position of the maximum is related to an activation energy which describes thermal diffusion of flux lines. In order to account for the width of the maximum a distribution of activation energies has to be taken into account. This distribution may be attributed to the structural quality of the films.

Measurements at constant temperature and varying field exhibit spikes in the IF at particular values of the field. There are indications that these spikes are related to matching effects of the flux lattice with respect to the geometry of the sample thus providing a means to investigate the structure of the flux line lattice.

**P5-14**  
**AMPLITUDE DEPENDENT DAMPING AND YOUNG MODULUS DEFECT OF COARSE GRAINED Y-Ba-Cu-O CERAMICS AT 6-20K**

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Amplitude-dependent internal friction and Young modulus defect of  $\text{YBa}_2\text{Cu}_3\text{O}_y$  ( $y=6.89$ ) are investigated by composite oscillator technique at frequency of longitudinal vibrations of about 100 kHz at temperatures from 6 to 20K. It is found that irreversible changes of the acoustic parameters (decrement and resonant frequency of the sample) which arise during measurements of their amplitude dependencies at 6K are fractionally disappeared during low temperature annealing up to 20K. The annealing up to room temperature recovers the values of acoustic parameters completely. It is shown that magnetic field up to 1.5T does not influence on measured acoustic effects. The data are discussed in the framework of a model where dislocation interacts with some pinning centers mobile at very low temperatures.

P5-15  
STRUCTURE TRANSITIONS AND INTERNAL FRICTION OF HIGH  $T_c$  SUPERCONDUCTORS

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Effect of structural defects on the elastic moduli, dissipative properties and acoustic emission of the high  $T_c$  ceramics and monocystals of  $YBa_2Cu_3O_{7-\delta}$  with  $\delta$  varying from 0 to 1 was investigated in the temperature range from 4.2 K to 300 K. The measurements were carried out at the frequencies of 5 MHz and 100 kHz by high frequency resonance and composite vibrator methods, respectively. The measurements of acoustic emission were made at frequency of 2 MHz. It was found that the temperature spectra of internal friction for the superconducting ( $\delta=0$ ) ceramic specimens contained several peaks at the temperatures of 80, 110-150 and 240 K. Simultaneous measurements of the elastic modulus revealed strong hysteresis behavior during heating and cooling processes. For the specimens with  $\delta>0.5$  the internal friction temperature spectra were different. In all cases the peaks magnitudes and the depth of hysteresis curve were strongly dependent on the quality of the specimens (by presence of voids, cracks, by perfection of intergrain contacts). It was also observed that during the heating and cooling of ceramic samples acoustic emission signals appeared at the temperatures which corresponded to the internal friction peaks temperatures. The measurements carried out with single crystalline specimens of the same composition showed that the level (intensity) of acoustic emission signals were essentially lower than for ceramic samples. The results observed are discussed in terms of the conception of structural rearrangement of the intergrain region due to temperature.

P5-16  
THE DEPENDENCE OF FLL DISSIPATION ON MAGNETIC FIELD ORIENTATION IN CERAMIC HIGH- $T_c$  SUPERCONDUCTORS

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With the vibrating reed technique, we have studied the flux-lattice-line (FLL) dissipation of ceramic YBCO and BSCCO as a function of transverse magnetic field with different relative angle between magnetic field and the vibrating direction of sample. The modulus (M) and internal friction (IF) increase with the applied field and their response to the magnetic field varies with the angle. When the angle is 90°, no significant change of M and IF with magnetic field is observed. With the angle decreased, the response of M and IF to field increases and gets to its maximum at 0 angle. Different deformations of FLL in different orientation of field due to the anisotropy of elastic constants of FLL were used to explain the observed phenomena.

**P5-17**

**THE ANOMALOUS FREQUENCY DEPENDENCE OF ENERGY DISSIPATION IN Bi-V-Sr-Ca-Cu-O SUPERCONDUCTORS**

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We measured the ac complex susceptibility of sintered  $\text{Bi}_{1-x}\text{V}_x\text{Sr}_2\text{CaCu}_2\text{O}_8$  and  $\text{Bi}_{1-x}\text{V}_x\text{Sr}_2\text{CaCu}_2\text{O}_8$  as a function of temperature (60 - 150K) and ac magnetic field amplitude (1-10Oe) and frequencies (10 - 500Hz). According to our investigation, the imaginary part of the susceptibility  $\chi''$  corresponding to power loss depends on the measuring frequencies strongly. The loss peaks shift from 78K to 94K with the frequency increasing from 10Hz to 100Hz, and shift from 94K to 77K with the frequency increasing from 100Hz to 500Hz. Only flux creeps model fail to explain that the peaks of energy loss shift with temperature as frequency  $f$  increases.

A model considering Anderson flux creeps and current density in critical state accounts for the observed frequency dependence, the discussion will give the pinning force quasi-quantitatively in intergranular vortices of polycrystalline High-Tc superconductors.

**P5-18**

**INTERNAL FRICTION STUDY OF 2201 PHASE IN BiSrCaCuO SYSTEM**

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Internal friction measurements were carried out using the flexure bending and torsional pendulum techniques on pure and dopping polycrystalline specimens of Bi-2201 phase in different stoichiometric conditions. An large acoustic frequency internal friction peak can be observed around 150K. The relaxation strength depends considerably on the amount of the oxygen vacancy in the specimens, which increases with increasing the amount of the vacancy in the specimens. We suggest that this internal friction peak is caused by the migration of the oxygen vacancies in the Sr-O plane. In the range of 90 to 130K, there exists a low frequency internal friction peak associated with the anomalous modulus in La dopping Bi-2201 specimen. It is a phase transformation internal friction peak. In the paper the results of ultrasonic velocity obtained from the specimen of pure Bi-2201 phase is also reported.

PS-19  
ULTRASONIC STUDY ON HIGH- $T_c$  SUPERCONDUCTOR  $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$

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Ultrasonic measurements on high- $T_c$  superconductor LSCO polycrystalline samples have been performed with temperature from 4 to 300 K. Two attenuation peaks are observed around  $T_c$  in these samples. As the Sr concentration increases, the one below  $T_c$  shifts to  $T_c$  until reaches it, the other above  $T_c$  shifts from 106 to 78 K as the doping level varies from 0.1 to 0.21. Except for the lattice softening caused by the T-O phase transition an additional softening tendency exists near 40 K, a hardening tendency is existed near 80 K. The lattice softening phenomenon disappears after the superconducting phase is formed. As Sr concentration increases the two tendencies become blur, the softening phenomenon becomes more and more apparent and the Poisson's ratio of the sample increases in the mean time, but the Debye temperature is anticorrelated with  $T_c$  in these samples, Which shows the same feature as the BCS superconductors.

PS-20  
NEW POSSIBILITIES IN ULTRASONIC STUDIES OF THE PHONON SPECTRUM FROM HIGH- $T_c$  SUPERCONDUCTING CRYSTALS

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Based on the phenomenological theory of sound propagation in anisotropic crystals, the authors propose a method of solving the main problems of the acoustic spectroscopy of high- $T_c$  superconducting crystals, namely, determination of characteristics and properties of soft phonon modes interacting with acoustic waves. The information gained with this method is an essential addition to the data of neutron diffraction and optical studies of the phonon spectrum from high- $T_c$  superconducting crystals. On the basis of crystal symmetry properties, the theory predicts most informative crystallographic direction for the phonon spectroscopy, e.g., for the tetragonal crystal with a perovskite structure these are the [100] and [110] directions.

The theory has made it possible to explain the experimentally observed anomalies of the temperature dependences of the velocity  $V_n(T)$  and the sound absorption coefficient  $\alpha_n(T)$  in  $\text{La}_2\text{CuO}_4(\text{Li}_x)$  single crystals ( $n=[001], [100], [110]$ ), which are due to both the thermal excitation of soft optical modes and phonon spectrum rearrangements in the vicinity of the structural phase transition.

For the crystals under study, determined are the type, symmetry and eigenfrequencies  $\omega_0$  of soft modes, as well as the character of unit-cell deformation during the structural phase transition. It is shown that soft-mode atom vibrations are, in fact, two dimensional, and the implantation of Li impurity into the  $\text{La}_2\text{CuO}_4$  lattice results in the change of the soft-mode dispersion law parameters. It is also shown that the singular part of the sound-velocity anisotropy parameter in the basal plane is about  $1/\omega_0^2 \min$ , and this allows direct determination of the temperature dependence  $\omega_{0 \min}$  of the soft mode.

P5-21

ULTRASONIC ATTENUATION TEMPERATURE BEHAVIOUR IN YBCO POLYCRYSTALS AND ITS POSSIBLE CORRELATION WITH THE HYSTERESIS LOOPS IN VELOCITY MEASUREMENTS

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Polycrystalline  $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$  ( $x > 0.9$ ) samples with various density values were investigated. Ultrasonic attenuation and velocity measurements for longitudinal and shear waves were carried out using broad band transducers with central frequencies at 20 MHz, 10 MHz and 5 MHz, in a standard pulse-echo configuration.

Attenuation measurements in the range 50 K to room temperature are presented. The data show a broad peak centered at about 230 K and a less pronounced peak at lower temperature that can be related to two hysteresis loops observed in the velocity vs. temperature curves. These results are discussed and compared with previous works on the subject.

P5-22

ANALYSIS OF THE OXYGEN RELAXATION SPECTRUM IN  $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$

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It is well known that the superconducting  $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$  ceramics exhibit an energy dissipation peak at the temperature around 470K ( $f = 1.0\text{Hz}$ ). This peak is a relaxation peak, with an activation energy  $E = 1.1\text{eV}$  and a pre-exponential factor  $\tau_0 \sim 10^{-13}\text{sec}$ . It can be attributed to the diffusion of the oxygen atoms within the Cu-O basal planes. However, the relaxation mechanism exhibits a behaviour which is not classical. Effectively, the dependence of the peak height on the oxygen concentration is not regular. Therefore, this peak can not simply be explained by a simple point defect relaxation.

In the present work, we present a systematic study of the evolution of this relaxation peak as a function of the oxygen concentration ( $x$ ). It is observed that the height of the peak varies dramatically as the oxygen concentration is changed. The variation of the peak height can be explained by a two dimensional model. Moreover, the shape of the peak is asymmetrical and therefore can not be interpreted by a single relaxation mechanism. Instead, the peak can be dissociated into two enlarged Debye peaks which are controlled by an energy distribution. This way of decomposition of the observed peak is more meaningful by taking into account the structural transformation which is related to the variation of the oxygen content in the specimen.

**P5-23**  
**ACOUSTIC ABSORPTION IN HIGH-TEMPERATURE SUPERCONDUCTORS DUE TO PSEUDO**  
**JAHN-TELLER CENTERS**

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The temperature dependence of the acoustic absorption of  $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$  and  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  up to 100 K is calculated using the two-well tunneling systems theory. The presence of tunneling systems in high- $T_c$  superconductors due to Pseudo Jahn-Teller effect, which specifies the tunneling systems' parameters, is the main assumption in our calculations. The relaxation times related with the microprocesses of the absorption are determined by the reaction rate method. The relaxation processes taking into account in our calculations are the non-radiative transitions as well as phonon assisting tunneling. The obtained results are in a good agreement with the experimental measurements. The present calculation supports our assumption for the presence of tunneling systems originated from Pseudo Jahn-Teller effect in high- $T_c$  superconductors.

**P5-24**  
**SURFACE ACOUSTIC WAVE ATTENUATION IN SUBLIMATED  $\text{C}_{60}$  THIN FILMS**

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The fullarene( $\text{C}_{60}$ ) thin films ( $\sim 500$  nm thick) were deposited onto the SAW (Surface Acoustic Wave) devices (lithium-niobate and quartz substrates) directly between the generating and the receiving inter-digital-type electrodes by sublimating the fullarene powder at 300 °C, and the temperature dependence of the SAW attenuation was measured at temperatures between 70 K and the room temperatures, and at 200 MHz for lithium-niobate and 120 MHz for quartz. It was demonstrated that this method is quite effective to investigate such materials composed of mesoscopic particles by weak Van der Waals force.

- The experimental results are so far summarized as follows.
- (1) Sharp attenuation peaks were observed near at 200 K and 220 K for the film deposited onto lithium-niobate substrate.
  - (2) Broad attenuation peak was observed (compared with that for lithium-niobate) near 180 K for quartz substrate. Namely, the behavior of the temperature dependence of the attenuation depends on the substrate.
  - (3) The temperature dependence curves showed hysteresis between the rising sweep and the reducing sweep in both cases.

P5-25

INTERNAL FRICTION IN Bi-Sr-Ca-Cu-O GLASSES

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The results of internal friction and DC conductivity measurements in  $\text{Bi}_2\text{O}_3$ -CaO-SrO-CuO glasses and partly crystallized glasses were reported. A comparison has been made with internal friction spectra of superconducting ceramics and copper phosphate glasses.

The internal friction measurements were performed by low frequency vibration reed and torsion pendulum methods. The mechanical loss peak observed at 150K (at 200 Hz) in crystallized glass and bismuth superconducting ceramics can be correlated with the mechanism of the DC conductivity in crystallized glass. The similarity of the relaxation parameters suggests that the same mechanism is responsible for both processes. It may be associated with the charge transfer between crystalline granules. In glassy state the DC conductivity activation energy is higher (0.6eV). It suggests that the small polaron hopping between copper ions in a different valency state  $\text{Cu}^{1+}$  -  $\text{Cu}^{2+}$  is responsible for charge transport in these glasses.

Copper phosphate glass exhibits a mixed electronic-ionic behaviour. A large internal friction peak in the high temperature range can be related to  $\text{Cu}^{1+}$  ion migration in the glass matrix. The high value of activation energy ( $\approx 1\text{eV}$ ) observed in DC conductivity indicated that ionic transport prevails in copper phosphate glass.

P5-26

ELASTIC AND RELAXATION PROPERTIES OF CuO AND HTS METAL-OXIDES

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It is known that the most of properties of the HTS metal-oxides are governed by the state of the cuprate planes Cu-O. The similar planes with a planar coordination of copper by oxygen ions in a monoclinic unit cell are present in CuO. In this work, the absorption spectra and velocity of ultrasound have been studied at temperatures 5 - 320 K and frequencies 47-370 kHz to establish common and different acoustic properties of the ceramics CuO, orthorhombic and tetragonal  $\text{YBa}_2\text{Cu}_3\text{O}_x$ ,  $\text{Bi}_{2.3}\text{Sr}_{1.7}\text{CuO}_{6.33}$ ,  $\text{Tl}_2\text{Ba}_2\text{CaCu}_2\text{O}_x$ .

The temperature spectra of sound absorption in CuO and HTS metal-oxide are similar in the intervals 30-60 K and 235-250 K. They have low- and high-temperature peaks the behaviour of which is similar for all materials when varying the vibration frequency. It is supposed that the high temperature peak is due to the antiferromagnetic ordering. Further studies are needed to clear up the nature of the low-temperature peak.

The greatest differences between the absorption spectra in the ceramics studied take place in the intermediate temperature range 100 - 200 K. The possible reasons of it are discussed in detail.

At thermocycling, the temperature dependences of sound velocity in CuO ceramics show a broad hysteresis analogous to that in HTS metal-oxides. The presence of the hysteresis in CuO and the tetragonal  $\text{YBa}_2\text{Cu}_3\text{O}_x$  does not agree with the assumption of its twinning nature. The development of a domain structure of the low-temperature phase below the transition point may be considered as a mechanism responsible for the hysteresis.

ACOUSTIC PROPERTIES OF  $\text{YBa}_2\text{Cu}_3\text{O}_x$ -Ag COMPOSITES

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The study of acoustic properties and structure of the composition  $\text{YBa}_2\text{Cu}_3\text{O}_x$ -Ag is made in a wide range of silver concentrations ( $c=0-100\%$ ).

The analysis of the behaviour of the structure sensitive ultrasound absorption peak shows that up to  $c = 20\%$  vol.% the substitution of the Cu atoms by the Ag ones does not take place and no changes in oxygen content are found.

In the samples with  $c \geq 10\%$  vol.% Ag a new relaxation peak the amplitude of which increases when increasing  $c$  is found. The activation parameters of this peak are determined and it is shown that the thermoactivated kink-pair formation on dislocations in silver is the most likely relaxation process responsible for this peak.

At thermocycling, the wide hysteresis is observed in the temperature dependences of sound velocity. When increasing  $c$ , the hysteresis has a tendency to decrease and disappears completely in the composite with  $c \geq 50\%$  vol.% Ag. It is obvious that the presence of plastic silver between grains provides relaxation of the thermoelastic stresses arising at thermocycling and removes the origin of the hysteresis.

For the first time, the concentration dependence of the longitudinal sound velocity is obtained and the non-monotonic character of the dependence is established in this composition. It is shown that formation of the intergrain silver layers improves mechanical contacts between grains at low  $c$  and leads to the increase of sound velocity. Its further decrease at higher  $c$  is due to a thickening of the layers of silver which has the lower sound velocity than the grains of  $\text{YBa}_2\text{Cu}_3\text{O}_x$ .

ULTRASONIC INVESTIGATION ON Na-DOPED Y-1-2-3 HIGH  $T_c$  SUPERCONDUCTORR. Ravinder Reddy, B.V. Reddi<sup>1</sup>, P. Venugopal Reddy

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## ABSTRACT

$\text{Y}_{0.9}\text{Ba}_{1.9}\text{Na}_{0.1}\text{Cu}_3\text{O}_x$  high  $T_c$  superconductor was prepared by the Solid-state reaction method using highly pure (99.9%)  $\text{Y}_2\text{O}_3$ ,  $\text{BaCO}_3$ ,  $\text{NaCO}_3$  and  $\text{CuO}$  taken in stoichiometric ratio of 1:2:3. The calcined powder after grinding and pelletizing was sintered at  $965^\circ\text{C}$  for 50 hrs. in air.

The ultrasonic longitudinal velocity measurement was carried out by the pulse transmission technique over a temperature range 80-300K. The velocity after remaining almost constant between 300-255K, is found to increase rapidly reaching a maximum value of 5600m/s at about 125K. On further cooling the velocity, however, is found to decrease continuously reaching a minimum value of 2000m/s at 90K. In the superconducting phase the velocity is found to remain constant. The observed anomalous longitudinal velocity behaviour may be explained qualitatively on the basis of a model. According to this model, such maxima are likely to occur at temperatures  $T \approx 2T_c$  due to the instability of the lattice and are likely to disappear with the addition of dopants like sodium, potassium and lithium etc.

P5-29

DAMPING OF THE ELASTIC MODULUS TEMPERATURE HYSTERESIS AND OF THE 230 K ACOUSTIC LOSS PEAK WITH DECREASING THE GRAIN SIZES IN HTSC YBaCuO

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Temperature dependencies of the Young's modulus and of the acoustic losses in HTSC  $\text{YBa}_2\text{Cu}_3\text{O}_x$  samples of different microstructures (grain sizes 1 - 50  $\mu\text{m}$ ) were studied. Measurements were carried out using a 130 kHz composite vibrator in the temperature range of 4.2-300 K. The samples were prepared using a new method based on the preliminary mechanochemical oxide activation. This method allows to vary the grain sizes in a wide range (1  $\mu\text{m}$  and even smaller in the super-fine-grained samples). The  $T_c$  value, the phase state, the grain sizes, and the intragrain structural nonuniformity were controlled for all the samples investigated.

In the coarse-grained samples, the temperature hysteresis of the Young's modulus is often observed which is accompanied by the acoustic loss peak near 230 K. These effects dampen with decreasing the grain sizes, and they completely vanish in the super-fine-grained samples.

These findings are interpreted in terms of the first and the second-order phase transitions, according to the  $T,x$ -diagram appropriate for  $\text{YBa}_2\text{Cu}_3\text{O}_x$ . Theoretical treatment of thermodynamics of the oxygen ions ordered along the Cu1-O4 chains shows that the interphase boundaries on the  $T,x$ -diagram strongly deform, provided that the intragrain local strains are taken into account when calculating the system's free energy. Some phases can even disappear at all. This mechanism may be responsible for dampening the structural phase transformations in the super-fine-grained samples, and, thus, for vanishing the hysteresis and the acoustic losses in such samples.

P5-30

ULTRASOUND PROPERTIES OF  $\text{C}_{60}$  FILMS

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Attenuation of acoustical surface waves in the range 100-500MHz has been measured in sublimed  $\text{C}_{60}$  films. A large attenuation governed by thermally activated mechanisms having activation energies of 0.07 and 0.3 eV and due to molecular reorientations is observed between 100 and 300K.

**P5-31**  
**ULTRASONIC STUDY OF THE EFFECT OF Ag DOPING ON THE METASTABILITY OF Gd-Ba-Cu-O SUPERCONDUCTORS**

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Ultrasonic velocity as well as attenuation have been measured as a function of temperature in Ag doped  $\text{GdBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (Gd123) systems. The systems studied are  $\text{GdBa}_2(\text{Cu}_{1-x}\text{Ag}_x)_3\text{O}_{7-\delta}$  with  $x = 0.0, 0.01, 0.02, 0.03$ . The samples have been prepared by the solid state reaction of stoichiometric mixture of  $\text{Gd}_2\text{O}_3$ ,  $\text{BaCO}_3$ ,  $\text{CuO}$  and  $\text{Ag}_2\text{O}$ . Resistivity as well as oxygen content have been determined in these samples. Ultrasonic measurements have been carried out in all the four samples in the temperature range 85-300K using Pulse Echo Overlap (PEO) technique. The relative velocity and attenuation have been determined in all the samples. The velocity data shows anomalous increase around  $T_c$  and a broad peak around 210K. The broad peak around 210K in the pure sample is found to shift with Ag doping. It is attributed to the oxygen ordering occurring in the Cu-O basal plane due to the metastability of the 123 system. Room temperature resistivity and the  $T_c$  values are found to support these results. The ultrasonic attenuation also shows a similar type of behaviour.

**P5-32**  
**THE ULTRASONIC PROPERTIES OF DIFFERENT SUPERCONDUCTING PHASES IN BiSrCaCuO CERAMIC**

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Ultrasonic longitudinal velocity and attenuation of superconducting phases 2201, 2212 and 2223 in BiSrCaCuO ceramic have been measured with pulse echo technique at 5MHz and 10MHz, between 15K and 300K, cooling and warming. The results display a sharp elastic transition near 250K for each sample. This transition shows significant thermal hysteresis for 2212 and 2223 phase sample (20-30K), but only a small thermal hysteresis to 2201 phase sample. The reason is discussed in this paper. In Pb-doping  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$  sample, we found a new ultrasound anomaly at about 170K. We suggest that it is caused by a kind of Martensitic phase transition.

# P5-33

## INTERNAL FRICTION RELATED TO CARRIERS IN HIGH-T<sub>c</sub> SUPERCONDUCTORS

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For both BiSrCaCuO and TlBaCaCuO samples, internal frictions ( $Q^{-1}$ ) in the kHz range reveal a plateau ( $Q_p^{-1}$ ) above  $T_c$  and drop rapidly below  $T_c$  with the turning points locating just at  $T_c$  for various samples of BiSrCaCuO and TlBaCaCuO with different  $T_c$ . This anomaly can not be observed for non-superconducting samples. Moreover, it is discovered that the  $Q_p^{-1}$  is nearly proportional to Hall carrier density for Y(Pr)BaCuO with various Pr content. These results show that the drop of  $Q^{-1}$  below  $T_c$  is closely related to superconducting condensation. The mechanism of  $Q_p^{-1}$  may be attributed to the stress-induced reorientation of the polaron-like distortion with various effective variants around the carriers. Further, with taking account of the smearing of superconducting gap structure resulting from the recombination of quasi-particles and by modifying the BCS relative jump rate as  $S(E, E', \Gamma) = \text{Re} \{ 1 - \Delta^2 / [(E - i\Gamma)(E' - i\Gamma)] \}$ , the calculated results of internal friction below  $T_c$  are in agreement with the experimental data fairly well. The superconducting gap  $\Delta$  and the damping rate  $\Gamma$  for both BiSrCaCuO and TlBaCaCuO have also been obtained, they are pretty in accordance with those got by tunneling spectrum and NMR methods etc.

# P5-34

## ULTRASONIC ATTENUATION NEAR $T_c$ IN HIGH-T<sub>c</sub> TlBaCaCuO SUPERCONDUCTORS

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Ultrasonic attenuation of good TlBaCaCuO samples reveals a plateau above  $T_c$  and drops rapidly below  $T_c$  with turning point locating at  $T_c$ , but the decrease is not so steep as that of conventional superconductors. Comparing our results with other experiments for high- $T_c$  superconductors, it can be concluded that this anomaly is closely related to the superconducting condensation. Further, with taking account of the smearing of superconducting gap structure resulting from the recombination of quasi-particles and modifying the BCS relative jump rate as  $S(E, E', \Gamma) = \text{Re} \{ 1 - \Delta^2 / [(E - i\Gamma)(E' - i\Gamma)] \}$ , the calculated results of attenuation are in agreement with the experimental data fairly well. The superconducting gap  $\Delta$  and the damping rate  $\Gamma$  for TlBaCaCuO have also been obtained, they are pretty in accordance with those got by tunneling spectrum and NMR methods etc.

**P5-35**  
**RELATIONSHIP BETWEEN INTERNAL FRICTION AND MAGNETIC FLUX PINNING FORCE**  
**OF  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  SUPERCONDUCTORS**

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Internal friction of magnetic flux pinning of bulk and film YBCO superconductors was measured in temperature range of 10 to 150K and magnetic field 0 to 1.5T. In small vibrating amplitude range, the internal friction increases with the decreasing of temperature which is far away from  $T_c$ , and the internal friction of film specimen is larger than that of bulk specimen. This behavior shows that the larger the pinning force, the larger is the internal friction. The possible reason is discussed in the paper.

**P5-36**  
**INFLUENCE OF LOW MAGNETIC FIELD ON ULTRASONIC ATTENUATION OF HIGH  $T_c$**   
**SUPERCONDUCTOR**

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Recently, a result about the influence of high magnetic field, at 40, 80, and 120KG, on the attenuation of ultrasonic wave with low frequency for BYCO superconductor has been present by Pantert. The influence of high magnetic field at a few thousand gauss and a few Tesla on attenuation of ultrasonic wave with high frequency (a few hundred MHz) for YBCO has been reported by levy. The influence of low magnetic field on the attenuation of ultrasonic wave with low frequency for YBCO at liquid nitrogen(LN) temperature was presented here. The attenuation of ultrasound was examined by changing the orientation between magnetic field  $H$  and ultrasonic wavevector  $q$  and using pulsed ultrasonic transmission method. The ultrasonic attenuation depends on the magnitude and orientation of magnet field. After magnetic field turning on, the sound amplitude at first increases with the increasing of magnetic field. When  $H$  reaches 250G (we regard it as  $H_{c1}$ ), the sound amplitude decreases rapidly and gets saturation value. The process from magnetic field turning on destroying superconductivity have a delay. That means time is needed for the influencing of magnetic field. Discussion is focused on the possibility of the relation between the ultrasound attenuation and the flux lattice melting.

P5-37  
A REVIEW ON THE ULTRASONIC BEHAVIOUR OF SOME HIGH  $T_c$  SUPERCONDUCTORS  
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The author as a Principal Investigator of a Research Project on High  $T_c$  Superconductors and his students have been investigating for the last five years, the ultrasonic longitudinal velocity & attenuation behaviour of number of polycrystalline ceramic superconductors mainly the Rare-earth 1-2-3, Bi-2212, 2223, Bi-Pb-2223, Y-1-2-3/Ag composites etc; having different compositions with varying concentration of dopants prepared by the Solid state reaction / Sol-gel routes over a temperature range 80-300K. The study is desinged to understand the physics of elastic behaviour of the new class of oxide superconductors. A review of the work done so far, covering various aspects of the investigation, is given here.

The ultrasonic longitudinal velocity of most of the samples in contrast to the normal solids is found to decrease with decreasing temperature in the temperature region 200-100K. For some samples, the decrease of velocity is unprecedented and is as high as 70% of the room temperature value. On further cooling, the velocity after showing a slight increase, however, is found to remain constant in the superconducting phase. The anomalous behaviour of the longitudinal velocity, which generally starts at temperature  $T \approx 2T_c$ , predicts that the lattice of the material is in an instable phase over a very long temperature range of about 100K. The anomalous variation of  $V_l$  has been explained as due to the weak links or low intergrain contacts. The coarse grains of the sintered samples are also may be responsible for the observed behaviour. The ultrasonic attenuation data of most of the samples yielded three peaks at temperature  $\approx 250K$ ,  $\approx 150K$  and 100K. A model to explain the observed peaks qualitatively is given

P5-38  
THE SAW VELOCITY CHANGE IN  $C_{60}$  FULLERITE FILM  
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In this article, the velocity change of surface acoustic wave (hereafter SAW) propagating along  $C_{60}$  fullerite film, measured using pulsed SAW technique was presented. The  $C_{60}$  fullerite films are deposited on Crystal of Y-Z  $LiNbO_3$  and 127.86 Y-X  $LiNbO_3$  substrates by thermal sublimation respectively. It is observed that the SAW velocity change depends on the temperature, the thickness and the film quality. Two discontinues changes of SAW velocity were observed at about 253k and 133k for mixture fullerite film. The former is attributed to motion of interphase boundary of the order-disorder transition. The phase transition at 133K may be related to superstructure of the film. Only one discontinue change of SAW velocity was observed about 184 K for pure and annealing film. The mechanism of this phenomena appearance is discussed. The comparison with other results obtained at low frequency by other authors by means of the modified vibrating-reed technique and free-free bar apparatus is given. The principle and result about the how to determine the film thickness from SAW velocity are given too.

# P6-1

## LOW TEMPERATURE ACOUSTIC ATTENUATION IN LANTHANIDE PHOSPHATE GLASSES

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The temperature behavior of the acoustic attenuation in  $\text{La}_2\text{O}_3\text{-P}_2\text{O}_5$  and  $\text{Sm}_2\text{O}_3\text{-P}_2\text{O}_5$  glasses shows the effect due to thermally activated relaxations of structural defects in the 15K-400K range and to phonon-assisted relaxation of two level systems (TLS) in the 1.5K-15K range. The addition of lanthanide ions decreases the relaxation strengths for both the TLS phonon assisted and thermally activated relaxations. This implies that the same centres are responsible for both the tunneling and high temperature classical relaxation effects. The average activation energy of the process does not show significant variations with increasing concentration of lanthanide ions. This finding strongly suggests that the local arrangement of the relaxing particles is not greatly influenced by the addition of of the network modifier ions.

Taken together these two observations support the conclusion that the relaxing particles are in the phosphate skeleton. Since the skeleton of the vitreous phosphate is constructed from three corner bonded tetrahedra ( $\text{PO}_4$ ) and the addition of modifier reduces the number of bridging oxygens, it is argued that two types of models can be used to describe the relaxation mechanism: the local motions of the oxygens which bridge between neighbouring tetrahedra or the harmonic libration of the coupled  $\text{PO}_4$  tetrahedra.

# P6-2

## RELAXATIONS CORRELATION MODEL APPLIED TO AMORPHOUS MATERIALS

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Statistical distributions of relaxation times are classically used to explain width and shape of internal friction peaks. On the other hand, serial mechanisms or interactions between defects explain complex relaxations observed in crystalline solids. In amorphous materials, relaxations are often characterized by very large peaks and by aberrant values of the activation energy and the time constant. If correlative or hierarchic processes have been developed to understand such effects, their direct use to analyse relaxations is not yet realized.

We propose here a new phenomenological model of correlation between several relaxing defects. The main idea is that a first kind of defect can only relax when (a) second kind defect (s) is (are) in a preferential geometrical configuration in the neighbouring free volume. This hierarchy leads to a distribution of relaxation times for the first kind of defect depending on the relaxation time associated with the second defect.

If  $E$  and  $E'$  are their virtual activation energies (non interactive case), the apparent energy deduced for the correlative relaxation varies between  $E$  and  $E + E'/2$  depending on the temperature. This analysis has been applied as an example to the relaxation observed at 400 K in the glassy part of a SiC-LAS composite.

We extend this model to a  $n$ -correlative phenomenon ( $1 < n < \infty$ ) where an extended defect relaxes (specific energy  $E$ ) in the presence of  $n$  pseudo-defects (specific energy  $E'$ ). The apparent energy is a function of  $E$ ,  $E'$  and  $n$  and depends on chosen correlation types. When completely developed, this model would be used to analyse relaxation peaks associated with  $\alpha$  and  $\beta$  transitions in polymers.

**P6-3**  
**INVESTIGATION OF THE STRUCTURAL CHANGES OF IRRADIATED POLYETHYLENE BY  
INTERNAL FRICTION**

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A Kê-type torsion pendulum has been used at temperatures up to 470 K to measure dynamic modulus and internal friction in an low density ( $0.91 \text{ g/cm}^3$  at 293 K)  $\gamma$ -irradiated polyethylene. The samples were irradiated in argon atmosphere by  $\text{Co}^{60}$ , at a dose rate of 20 kGy/day. The dose range was 0-300 kGy.

According to our results two groups of relaxation peaks appeared in temperature range of 230-370 K:

- the group of  $\beta$ -transition peak developed at about 270 K,
- the maximums of the peaks of  $\alpha$ -transition group are in the temperature range of 355-360 K.

The  $\beta$ -peak appeared in low density polyethylene is related to glass transition caused by the freezing in and out of the segmental movements. This transition peak has not been observed in the internal friction spectra of the structurally more ordered high density polyethylene.

The  $\alpha$ -peak is related to the lattice site movements in the crystalline domains of our samples.

**P6-4**  
**LOW TEMPERATURE INTERNAL FRICTION IN COLD ROLLED AND HYDROGENATED  
 $\text{Ni}_{60}\text{Nb}_{40}$  METALLIC GLASS**

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Cold rolling of  $\text{Ni}_{60}\text{Nb}_{40}$  metallic glass (MG) results in appearance of large IF peak in the range  $240 \leq T_{\text{peak}} \leq 280 \text{ K}$  (at  $f \approx 300 \text{ Hz}$ ). Characteristics of this peak are quite close to that in other cold deformed MG. Hydrogenation also leads to arising of IF peak ( $220 \leq T_{\text{peak}} \leq 240 \text{ K}$ ) which is almost completely analogous to that in rolled samples. It is discovered the phenomenon of MG hydrogen plastification which consists in sufficient plastic deformation during hydrogenation under small tensile load. It is argued that plastic deformation without external stress takes place also and it is a result of stress relaxation in regions of hydrogen accumulation. It is concluded that IF in hydrogenated samples is not an analogue of Snoek relaxation. The nature of IF peaks in rolled and hydrogenated samples has common origin and is determined by dislocation-like defects which arise on plastic deformation.

# P6-5

## LOW-ENERGY RELAXATION IN GLASS STRUCTURE OF $\text{SiO}_2\text{-GeO}_2$ MIXTURE

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The glass state, as often represented by  $\text{SiO}_2$  glass, has characteristic thermal and acoustic properties at low temperatures, i.e., the specific heat linear in  $T$  and the thermal conductivity proportional to  $T^2$  below 1 K, acoustic properties explained by the two level system (TLS) model around or below 1 K, and the large acoustic relaxation peak around the nitrogen temperature. These properties are generally considered to be due to the existence of double-well potential in some atomic configuration. However, there is no definite solution on the real atomic configuration for it until now.

Our approach for seeking the solution is very simple but may be appropriate, i.e., we have tried to measure precisely the ultrasonic absorption or internal friction (IF) of a mixed glass of 90 %  $\text{SiO}_2$  and 10%  $\text{GeO}_2$ , where the sites of Si atoms in  $\text{SiO}_2$  glass are expected to be replaced randomly by Ge atoms. The IF is measured by a composite oscillator method using - 18.5°X-cut quartz transducer (51 kHz).

We have found a large relaxation loss between 4.2 K and 200 K, which has a broad peak around 50 K. The loss is explained as the addition of the component observed in pure  $\text{SiO}_2$  glass which should have a peak around 35 K, and a new one which has a peak around 65 K and may come from the Si-O-Ge bridge in the mixed glass.

This result prefers that the most probable origin of the structural relaxation of  $\text{SiO}_2$  glass is transverse motion of the oxygen atom in Si-O-Si bridge among the existing structural models. In addition it is suggested that the similar mechanism is the origin of TLS phenomena below 10 K. Further study of the dependence on the composition rate in  $(\text{SiO}_2)_{1-x}(\text{GeO}_2)_x$  is in progress.

# P6-6

## ELASTIC AND UNELASTIC RELAXATION MECHANISMS IN MULTICOMPONENT SUPERIONIC PHOSPHATE GLASSES

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The acoustical velocity and attenuation are investigated by pulse-echo and acoustooptic techniques in  $(\text{M}_2\text{O})_x(\text{P}_2\text{O}_5)_y(\text{Nb}_2\text{O}_5)_{1-x-y}$  glasses, where M: Li, Na, K, Rb and  $X = (30 - 55)\%$ ,  $Y = (27,5 - 50)\%$ . The electrical conductivity of the glasses varied with the composition from  $10^{-8}$  to  $2.5 \cdot 10^{-8} \Omega^{-1}\text{cm}^{-1}$ . The measurements were made in wide ranges of temperature (10-400)K and frequency (14-1500) MHz. The techniques used made it possible to measure acoustic attenuation from 5 to 700 dB/cm. The results of our study can be summarized as follows.

1. It is shown that double-well potential system model is valid for description of both network defect and mobile ion dynamics in high temperature region.
2. The existing thermodynamical theory of acoustical interaction with two state defects is expanded.
3. The theory explained well the experimental data both for attenuation and velocity if: a) very wide distribution of network defect potential barriers and b) double Gaussian distribution for mobile ion double-well potentials are used.
4. It is found that ion double-well potential barriers are lower than that for mobile ion percolation.

P6-7

ULTRASONIC ATTENUATION MEASUREMENTS IN NEUTRON IRRADIATED QUARTZ: THE INFLUENCE OF HEAT TREATMENT FOR A DOSE OF  $4.7 \times 10^{19} \text{ n/cm}^2$ .

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In recent years, it has been well established that amorphous solids show low temperature properties which can phenomenologically be described by low energy tunneling excitations, the so-called tunneling states (TS). In order to contribute to the microscopic origin of these TS, we have been involved in ultrasonic studies of both electron and neutron irradiated quartz, since we could show that after irradiation, quartz reveals similar "anomalies" as amorphous solids.

Upon annealing, quartz irradiated with a neutron dose below a threshold of approximately  $6 \times 10^{19} \text{ n/cm}^2$  returns to the crystalline state. However, ultrasonic measurements after heat treatment for a dose of  $1.2 \times 10^{19} \text{ n/cm}^2$  revealed that there are still TS left.

Here, we report the results of the study of the TS after heat treatment extended to a higher dose: ultrasonic attenuation measurements are performed for a quartz specimen irradiated with  $4.7 \times 10^{19} \text{ n/cm}^2$  and annealed at  $700^\circ\text{C}$  during 1 hour. These measurements are carried out as a function of temperature (0.3 - 20 K) for different frequencies (80 - 300 MHz). A quantitative analysis to the tunneling model has been done and the derived parameters are compared with the results for this specimen before annealing and the parameters obtained after a similar heat treatment on a sample irradiated with the lower dose of  $1.2 \times 10^{19} \text{ n/cm}^2$ .

P6-8

ULTRASONIC ATTENUATION IN  $\alpha$ -QUARTZ DUE TO  $E'$  DEFECT CENTERS

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The ultrasonic attenuation in  $\alpha$ -quartz as a function of the temperature is calculated by taking into account the relaxation processes due to  $E'$  series defect centers. The relaxation processes involve  $E'_1 - E'_1$ ,  $E'_2 - E'_4$  interconversions and relaxation of the hydride ion at  $E'_4$  defect centers. The relaxation times used in the calculations are computed on the base on the Pseudo Jahn-Teller effect and reaction rate method. The obtained loss curves follow the general trends predicted by the relaxation theory. The present calculation may be useful in forecasting the response of quartz to irradiation with high-energy particles.

**P6-9**  
**RELATIVE VARIATION OF THE SOUND VELOCITY IN PMMA AT INTERMEDIATE LOW-TEMPERATURES**

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We have extended measurements of the relative sound velocity variation in amorphous polymethyl methacrylate from 4 to 77 K. Our experimental results of the temperature dependence of the velocity shift  $\Delta v/v$  at intermediate low-temperatures confirm its linear decrease with temperature. We also find that the temperature coefficient of the velocity shift  $\beta = \partial(\Delta v/v)/\partial T$  in glassy PMMA appears to be frequency-independent in the range from 5 to 50 MHz.

**P6-10**  
**ACOUSTIC PROPERTIES AND THERMAL EXPANSION OF Ge-As-Se GLASSES**

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We have measured the velocity and attenuation of longitudinal and shear ultrasonic waves (30 MHz) and thermal expansion within the temperature range of 80-300 K for  $\text{Ge}_x\text{As}_y\text{Se}_{1-x-y}$  ( $0 \leq x \leq 0.3$ ,  $0.1 \leq y \leq 0.4$ ) glasses. The elastic parameters (Young's modulus, shear modulus, Poisson's ratio and Debay's temperature) of alloys were computed using our data on density at room temperature.

The results are discussed in dependence on mean coordination number  $\langle m \rangle$  (where  $\langle m \rangle$  is calculated as  $\langle m \rangle = 2 + 2x + y$ ) in the framework of the model of chemical ordered continuous random network (COCRN). The values of  $\langle m \rangle$ , which correspond to compositions under study, are within the range of 2.1-3. The anomalous behaviour of elastic parameters in dependence on  $\langle m \rangle$  is observed for glasses with  $2.51 \leq \langle m \rangle \leq 2.78$ . The characteristic temperature regions in width of 10-30 K, where the behaviour of temperature dependences of ultrasonic wave velocities may be caused by phase transitions, are detected for all alloys. The dependence of temperature mean value, which correspond to such region, on  $\langle m \rangle$  is in agreement with the dependences of the elastic parameters on  $\langle m \rangle$ . The temperature dependences of attenuation of both ultrasonic wave velocities exhibit some distinctions within these temperature regions. The temperature dependences of relative lengthening of the samples display no monotone behaviour for all alloys studied. Their the most complex form is observed for glasses with  $2.4 \leq \langle m \rangle \leq 2.78$ .

Our results are not consistent with the COCRN model. They may be interpreted in the framework of cluster model of glass structure.

**P6-11**  
**DAMPING INSTABILITIES OCCURRING IN INTERNAL FRICTION SPECTRA OF  
FERROMAGNETIC AMORPHOUS ALLOYS AS A FUNCTION OF TEMPERATURE**

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Internal friction spectra of Fe-Si-B, Fe-Co-Si-B and Fe-Mo-Ni-B ferromagnetic amorphous alloys were drawn as a function of temperature between room and crystallization temperatures.

Internal friction evolutions exhibit various stages associated with structural modifications. In the low temperature range (25-100°C) a first stage corresponds to an internal friction level maintained at a constant value, then a second one is characterized by an internal friction increase approximately linear between 100 and 200°C. Above this temperature (200-400°C), a little and broad maximum is developed in relation to structural relaxation. A next step, in the internal friction evolution, is due to the ferro-paramagnetic transition. This phenomenon appears as a limit between low temperatures structural modifications and the high temperatures ones when crystallization takes place (400-600°C).

More original results are obtained when experiments are performed on a Kê pendulum, the specimen being submitted to a slight longitudinal stress. Indeed, in the low temperature and crystallization temperature ranges respectively, instabilities are detected in the damping curves, i.e. in amplitude against time curves. Instabilities observed at low temperatures cover the two first internal friction stages and consequently they are assumed to be associated with the release of internal stresses. At high temperatures, when crystallization occurs, new instabilities are observed which are related to internal stresses due to an "explosive" crystallization phenomenon. The experimental results are presented taking in account the possible effects of an applied magnetic field.

**P6-12**  
**ULTRASONIC ATTENUATION IN SUPERIONIC CONDUCTORS**

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The results of experimental investigations of ultrasonic attenuation and velocity in superionic conductors are presented in this communication. The temperature dependencies of ultrasonic attenuation reveal high relaxational peaks and corresponding to them velocity dispersion. Such behavior is determined by acoustoionic interaction. The piezoelectric type of interaction have been observed in piezoelectric materials:  $\text{Ag}_3\text{SbS}_3$ ,  $\text{Ag}_3\text{AsS}_3$ ,  $\text{AgPbSb}_3\text{S}_6$  single crystals and  $\text{Ag}_2\text{S} + \text{Ag}_3\text{SbS}_3$  polycrystalline system. In piezoelectric crystals the single temperature dependent relaxation time for fast ionic system was obtained from attenuation and velocity measurements for piezoactive ultrasonic modes. This time was supposed to be the Maxwell relaxation time of ionic electric conductivity. The attenuation peak heights were caused by the corresponding electromechanical coupling constants; the peak width was determined by activation energy of ionic conductivity. The another type of acoustoionic interaction was observed in non-piezoelectric ionic conductors: polycrystalline  $\text{Ag}_8\text{HgS}_2\text{J}_6$ ,  $\text{Ag}_{16}\text{J}_{12}\text{P}_2\text{O}_3$ ,  $\text{Ag}_6\text{J}_4\text{WO}_4$  systems and also in  $0.55\text{AgJ}-0.45\text{AgPO}_3$  superionic glass. In this case the observed ultrasonic relaxational attenuation maxima were caused by elastic wave modulation of chemical potential felt by mobile ions, i.e. "deformation potential" mechanism. In these materials the wide frequency spectrum for ultrasonic relaxation has been observed. It is shown that ultrasonic attenuation peak height depends on the concentration of the mobile ions in the material and also of the square of the ion-phonon coupling constant. The characteristic ultrasonic attenuation and velocity anomalies observed in the vicinity of the structural phase transition temperatures in investigated superionic conductors also are discussed.

**P6-13**  
**INTERNAL FRICTION BEHAVIOUR OF a-Pd<sub>80</sub>Si<sub>20</sub> DURING MULTIFOLD ISOTHERMAL ANNEALING**

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Thermal history is one of the important factors, that affects the structural relaxation of amorphous alloys. Internal friction behaviour of a-Pd<sub>80</sub>Si<sub>20</sub> during multifold isothermal annealings at different or same temperatures has been studied and internal friction values ( $Q^{-1}(t)$ ) vs. annealing time ( $t$ ) curves were obtained. The internal friction data obtained can be fitted by a stretched exponential expression, as follows:

$$Q^{-1}(t) = A \exp(-t / \tau)^{\beta} + B$$

it was found that pre-heat treatments affect the relaxation time  $\tau$  and exponent  $\beta$ .

P7-1  
ANELASTIC RELAXATION ASSOCIATED WITH THE INTERGRANULAR PHASE IN SILICON  
NITRIDE AND ZIRCONIA CERAMICS

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Mechanical spectroscopy measurements were performed in silicon nitride and zirconia ceramics, using an inverted torsion pendulum working with forced vibrations, in the temperature range between room temperature and 1600K. In both ceramic materials a steep modulus decrease is observed at high temperature, which announces the beginning of creep deformation of the material (microcreep). In parallel, the mechanical loss angle spectrum presents a relaxation peak, superposed to an exponential background.

In both materials, the principal mechanism of creep deformation in the low stress regime is believed to be grain boundary sliding. This is controlled by the viscosity of the intergranular layer present in these materials. In the case of the zirconia ceramics, analysis of the mechanical loss spectra assuming a grain boundary sliding model showed a good correlation with the results of creep tests performed in the same materials. In particular, the influence of the amount of the intergranular phase, the influence of the grain size and the apparent activation energy values as obtained by both techniques, are very close. In the case of the silicon nitride ceramics, a well resolved mechanical loss peak is observed occurring at 1300K for 1Hz. The associated activation parameters are unusually high (apparent activation energy of about 1000kJ/mol and preexponential factor of  $10^{40}$  to  $45$  s<sup>-1</sup>) suggesting that the relaxation mechanism could be related to the glass transition taking place in the intergranular phase. Effectively, a highly irreversible evolution of the mechanical loss spectra was observed, depending on the chemical nature of the grain boundary interphase and on thermal history. This evolution could be correlated with the crystallization respectively amorphization of the intergranular phase, which was also observed by transmission electron microscopy.

The above results indicate that ceramics which contain an intergranular phase exhibit anelastic relaxation phenomena associated with grain boundary sliding which could give valuable information on the low stress creep behaviour of these materials.

P7-2  
DISLOCATION INTERNAL FRICTION MECHANISMS IN PROPER AND IMPROPER  
FERROELECTRICS-FERROELASTICS

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It is known, the features of internal friction (IF) near the structural phase transitions, in a great extent, are due to the coupling between the macroscopic phase transition parameter and an external force. Really, the behavior of the IF peak near the Curie point ( $T_c$ ) may be explained in terms of the Landau-Khalatnikov relaxation mechanism at rather high frequencies or the fluctuation loss mechanism at ultralow frequencies only in those cases when an external alternating stress disturbs a spontaneous strain (order parameter) from the equilibrium state in ferroelectrics-ferroelastics. But our experimental investigations have shown the presence of pronounce IF peak near the Curie point for the such oriented samples, where the external stress was not coupled with the order parameter. In the case of improper ferroelectric-ferroelastic gadolinium molybdate crystal the IF peak height near  $T_c$  increases with a heating rate and decreases with a strain amplitude of a sample and an oscillation frequency. To explain these regularities a new loss mechanism which is attributed to the interaction between the interphase boundaries and dislocations taking into account the antiphase domain boundaries has been developed. For comparison the IF measurements of the proper ferroelectric-ferroelastic potassium dihydrogen phosphate crystal at various heating rates and strain amplitudes have made at the same frequency. Now the IF peak height increases with the strain amplitude increasing. We believe that IF peak is due to the kinetics of dislocations interacting with phase boundaries and point defects.

**P7-3**  
**THE RELATION BETWEEN INTERNAL FRICTION AND TENSILE CREEP DEFORMATION ON ALUMINA CERAMICS**

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The properties of internal friction and tensile creep deformation are compared on alumina ceramics for structural use. The investigated alumina has 99.9% purity and average grain size of  $6.2\mu\text{m}$ . Internal friction was measured with torsion pendulum type testing equipment, with increasing temperature up to  $1450^\circ\text{C}$ . The frequency dependence of the internal friction was tested in the range from 1.27 to 12.0Hz by changing the weight of the pendulum and the thickness of the specimen. Tensile creep deformation was measured with dead weight type creep deformation testing equipment and optical extensometer. Creep test was conducted at 1350, 1400 and  $1450^\circ\text{C}$ . Apparent activation energy was calculated from both of the internal friction and the creep deformation. Stress exponent was also calculated from the creep deformation.

Internal friction increases exponentially with increasing temperature, and it shows no characteristic peaks with temperature changing. Then, internal friction values of  $10 \times 10^{-3}$  and  $20 \times 10^{-3}$  were used to obtain apparent activation energy from the relation between the internal friction and the frequency. It was obtained as 500 to  $590\text{kJ/mol}$ . On the other hand, apparent activation energy obtained from the tensile creep deformation was 580 to  $690\text{kJ/mol}$ . Those are relatively close to the values from the internal friction. However, concerning the influence of the grain size to the creep deformation and the internal friction, they show different results. Grain size dependence is significant on the creep deformation, and the exponent for the grain size dependence was obtained as 2 to 4. However, grain size dependence was not clearly observed on the internal friction. Then, we estimate that the activation energies from creep deformation and internal friction represents different phenomena. As the creep deformation is followed by the creep fracture and the stress exponent is larger than 1, it is estimated that the activation energy obtained from the creep deformation is affected by the cavity formation at the grain boundary. On the contrary, we estimate that the activation energy obtained from the internal friction represents some damping mechanisms not at the grain boundary but in the grains, as internal friction shows less dependence to the grain size.

**P7-4**  
**ULTRASONIC AND DIELECTRIC STUDY OF THE FERROELECTRIC PHASE TRANSITION IN  $\text{KH}_2\text{AsO}_4$**

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The anomalous part of the ultrasonic velocity and the attenuation of longitudinal ultrasonic waves in  $\text{KH}_2\text{AsO}_4$  at a frequency of about 10 MHz was measured. The experiment was performed on ultrasonic waves propagating along the crystallographic axis (parallel and perpendicular to the ferroelectric axis in the temperature range around the ferroelectric phase transition at  $T \approx 95\text{K}$ ). The measurements were performed using a self designed apparatus to record the ultrasonic transit time.

Using dielectric measurements the data of the piezoelectrically excited mechanical resonances were used to evaluate the temperature dependence of the soft elastic shear constant. From the dielectric data the domain freezing was found to behave in a similar way as found in  $\text{KH}_2\text{PO}_4$ .\*

\* E. Nakamura, K. Kuramoto  
Resonance and Relaxational Dispersion around the Domain Freezing  
Temperature in  $\text{KH}_2\text{PO}_4$   
J. Phys. Soc. Japan **57**, 2182(1988)

INTERNAL FRICTION AND MODULUS OF NANOSTRUCTURED  $\text{SnO}_2$  AND  $\text{ZrO}_2$  MATERIALSXie Cunyi, Zhang LideInstitute of Solid State Physics, Academia Sinica,  
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A systematic investigation on internal friction (IF) and modulus of nanostructured  $\text{SnO}_2$  and  $\text{ZrO}_2$  with different particle size was carried out. It was found that the behavior of IF and modulus of nanostructured  $\text{ZrO}_2$  was different from that of nanostructured  $\text{SnO}_2$ . With increasing sintering temperature, the IF decreased for nanostructured both  $\text{SnO}_2$  and  $\text{ZrO}_2$ , however, an obvious IF peak was observed for nanostructured  $\text{SnO}_2$ . As to nanostructured  $\text{ZrO}_2$ , the modulus increased monotonously with increasing sintering temperature, but the modulus of nanostructured  $\text{SnO}_2$  increased firstly and then decreased with increasing sintering temperature. The detail discussion for the phenomena was performed.

INTERNAL FRICTION STUDY OF TRANSFORMATION DYNAMICS AND DISSIPATION  
FUNCTION IN CERAMICS  $\text{BaTiO}_3$ J.X. Zhang<sup>1</sup>, W. Zheng<sup>1</sup>, P.C.W. Fung<sup>2</sup>, K.F. Liang<sup>1</sup>

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The internal friction  $Q^{-1}$  and relative modulus  $(\delta/\delta_0)^2$  of pure and doped(semi-conductive) polycrystalline ceramics  $\text{BaTiO}_3$  were measured by a multi-function inverted vacuum torsion pendulum(with forced oscillating model). The temperature range, frequency range and the heating rate of measurement were 120K to 500K, 0.1Hz to 5Hz and 0.25K/min to 5 K/min respectively. Three IF peaks and three modulus' minimums corresponding to three phase transformations ( $C_{1v}/C_{2v}$ ,  $P_1$ ;  $C_{2v}/C_{3v}$ ,  $P_2$ ;  $C_{4v}$  (paraelectric) /  $C_{4v}$  (ferroelectric),  $P_3$ ) are observed. All the IF peaks show a viscoelastic feature and the nature of the first order PT. The peak temperature is independent to the frequency of measurement and the peak height increases with an increasing of  $(T/\omega)^2$ . A coupling coefficient  $\alpha$  of phase interface to the oscillating force is found to be  $\alpha(\omega) \sim \omega^{-1}$ . For pure  $\text{BaTiO}_3$ ,  $n_1 = 0.25$ ,  $n_2 = 0.025$ ,  $l_1 = 0.093$ ,  $l_2 = 0.185$  for doped  $\text{BaTiO}_3$ ,  $n_1 = 0.025$ ,  $n_2 = 0.190$ ,  $l_1 = 0.101$ ,  $l_2 = 0.204$  ( $n_1 > n_2$ ,  $l_1 \sim 0.5l_2$ ). The behavior of IF shows that it is caused by the motion of phase interface during the process of the first-order phase transformation.

An interaction model and a phenomenological theory are proposed to explain the characteristics of IF and a dissipation function of the first-order phase transformation in  $\text{BaTiO}_3$  is acquired.

**P8-1**  
**INFLUENCE OF THE CATION CHARGE ON THE MOLECULAR MOBILITY OF POLYMER ELECTROLYTES**

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Recent studies emphasized the relevant role of the cationic charge in determining the structural behavior of polyethylene (PEO)-salt complexes, a very promising class of polymer electrolytes for their quite high ionic conductivity. It has been established that solid solutions of PEO and salt with singly charged cation (NaSCN and KSCN) show a multiple phase structure, which tends towards a single crystalline phase at high content of salt (mol. fract. ~ 0.20). In contrast in the case of divalent cations the structure of the complexes becomes a single amorphous phase, probably as a result of intermolecular links established by the cation between neighboring chains. In both the cases a remarkable increase of  $T_g$  follows the addition of salt, the complexes with the divalent cation showing the highest elevation rate of  $T_g$  with increasing concentration.

The present study of the anelastic relaxations in PEO-Fe(SCN)<sub>2</sub> complexes has been made to clarify the effect of the cation charge on the molecular motions and on the structural phases that characterize the PEO-based polymer electrolytes. It has been found that the local and cooperative molecular motions characterizing the host polymer are strongly influenced by the addition of the salt, as a result of the interactions between the chains and the salt molecules, which ensure the formation of homogeneous complexes. The salt stiffens the host polymer and gives rise to a relevant enhancement of the non-crystallinity in the low concentration region (mol. fract.  $\leq 0.10$ ). At low temperatures the single  $\gamma$  relaxation, observed in pure PEO, is enhanced by the presence of the salt and its strength follows a behavior closely correlated to that of the primary relaxation. A quantitative analysis in terms of a parallel relaxation permits the underlying local motions to be ascribed to the amorphous fraction of the polymer.

**P8-2**  
**INFLUENCE OF MOISTURE ON SECONDARY RELAXATION IN THERMOSET-THERMOPLASTIC MATRIX COMPOSITE**

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Damping properties of composite samples with TGDDM-DDMe-PEI organic matrix and carbon fibers was studied between 100°K and 500°K.

Experiments were carried out with two apparatus : a Rheometrics DMA (anisothermal measurements (3°K/min) at 1 Hz) and a forced oscillation torsion pendulum (isothermal measurements between 10<sup>-4</sup> Hz and 100 Hz).

Results obtained by dynamical analysis (1 Hz) show the high sensitivity of primary  $\alpha$  (~ 500°K) and secondary  $\beta$  (~ 220°K) relaxations with sample moisture content. For soaked specimens a new peak appears at 360°K.

For increasing moisture content, the apparent maximum situated at about 220°K shifts to a lower temperature with a broader area. The difference between spectra corresponding to wet and dry samples exhibits a peak situated at ~ 185°K and probably corresponding to the water molecule interaction with the matrix.

## ANHARMONIC AND RELAXATIONAL EFFECTS IN NaSCN POLYMER ELECTROLYTES

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The behavior with temperature of the dynamic elastic modulus  $E'$  in pure poly(ethylene) oxide (PEO) and PEO-NaSCN solid blends (M is the alkali metal) for temperatures below and at the glass to rubber transition region is explained in terms of two physical contributions: the first is due to the anharmonicity of vibrational modes; the second to the  $\alpha_1$ - or primary relaxation which arises from the cooperative segmental motions of the polymeric chains in the amorphous phase of the polymer. The strengths of both the contributions shows a strong dependence on the degree of crystallinity of the polymer, which has been varied over a wide range by altering the composition. The highest values of the parameters describing the two mechanisms are observed when the addition of the metal salt makes the system completely amorphous.

An average relaxation time  $\langle \tau \rangle$  is obtained by the evaluation of the  $\alpha_1$ -relaxation in terms of the stretched exponential or Kohlrausch-Williams-Watts (KWW) function  $\phi(t) = \exp[-(t/\tau)^\beta]$ , where  $0 < \beta \leq 1$ . Its behavior with the NaSCN content shows that it results from the interchain interactions in the amorphous fraction of the polymer and from the relative amounts of the crystalline and amorphous phases.

It has been revealed that the storage at room temperature of these polymer electrolytes, over the time interval explored, causes variations in the anharmonic and relaxation properties of the samples, which are ascribed to changes in the relative amounts of the phases building up the structure. The behavior of the elastic and anelastic characteristics with increasing annealing time shows that the structure tends to a growing degree of crystallinity and that the crystalline regions constrain the cooperative segmental motions in the amorphous fraction.

## P8-4

## ANOMALOUS SCATTERING OF ULTRASONIC WAVES IN COMPOSITE MEDIA NEAR THE PERCOLATION THRESHOLD

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Scattering of elastic waves near the percolation threshold in disordered solids is strong enough to make invalid a perturbation theory method [1,2].

Nevertheless, asymptotics of an effective refractive index and elastic moduli have been calculated with the use of renormalization group in  $(4-\varepsilon)$  - dimensional space.

It turned out that there is the characteristic frequency

$$\omega_* \sim |p - p_c|^{(1-\gamma + \varepsilon/2)\nu} \quad \text{when} \quad \omega \gg \omega_*, \quad \text{excitations in the}$$

medium become fracton-like with the density of states  $\rho_f \sim \omega^{\delta-1}$  where

$\delta = d [1 + (\varepsilon - \gamma)/2]^{-1}$  is the spectral dimension. The latter equation is the Alexander-Orbach relation derived analytically.

The average phonon Green function for disordered two-atomic crystal is calculated near the percolation threshold [2].

1. A.L.Korzhenevskii, A.A.Luzhkov. Sov.Phys.JETP, v.70, p.395 (1990); ibid., v.72, p.295 (1991).
2. A.L.Korzhenevskii - in print.

P8-5

THEORETICAL AND EXPERIMENTAL EVALUATION OF ULTRASOUND ATTENUATION IN  
POROUS SOLID MATERIALS

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The role of ultrasound attenuation to characterise porous solids is currently being investigated in cancellous bone and hydrated cements.

Cancellous bone consists of a porous solid foam interspaced with bone marrow with porosities in the range of 70% to 95%. There is increasing interest in assessing the role of broadband ultrasound attenuation (BUA) in the identification and management of osteoporotic changes in cancellous bone. BUA is related to the density and structure of cancellous bone, although the exact dependence has not been determined to date. Complementary theoretical and experimental studies based upon the concept of permeability have been undertaken, providing an insight into the dependence of BUA on structure.

The onset of hydration in cement pastes induces a transition from a viscous suspension into a highly connected porous solid. The porosity of the cement is partly governed by the water content, but mainly by the addition of certain admixtures, such as air entraining agents. Hardened cement paste has been shown to exhibit a linear decrease in ultrasound velocity, in contrast to an increase in BUA, with increasing porosity in the range 10% to 50%.

P8-6

THE APPLICABILITY OF THE ARRHENIUS RELATIONSHIP ASSOCIATED WITH THE  $\alpha$ -PEAK  
OF POLYSTYROL

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By means of measurment of internal friction in amorphous polymer polystyrol, it was demonstrated that the  $\alpha$  internal friction peak has a derivation from the Arrhenius Relationship in form. More importantly, it was proved that the varying of activation energy with temperature led to the derivation.

P8-7

MODELLING AND NUMERICAL COMPUTATION FOR TRANSIENT INTERNAL DAMPING  
DUE TO THE THERMAL EXPANSION MISMATCH BETWEEN MATRIX AND PARTICLES IN  
M.M.C.

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A new model for transient internal damping (I.D.) associated with the emission and movements of dislocations around particles in metal matrix composites is developed. The proposed model is based on these movements that are mainly induced by the internal stress field around particles, which results from the thermal expansion mismatch between particles and matrix, during temperature variations.

First, from this thermally induced internal stress field, calculated by the ESHELBY method, and the critical stress  $\tau_f$  opposing motion of dislocations in their glide plane in the matrix, the number and the positions of the dislocations are determined as a function of temperature.

Second, the actual positions due to the superposition of the alternating shear stress associated with the pendulum oscillation are calculated by a perturbation method.

Then the internal damping is derived from the contribution of the dislocation movements to the inelastic strain over a period of oscillation. The role of the experimental parameters (cooling rate, frequency and amplitude of oscillation) and material characteristics ( $\tau_f$  evolution vs temperature) is investigated. This simulated I.D. is compared to the experimental results obtained in the case of aluminium based metal matrix composites (see paper Transient Internal Damping in Aluminium Based Metal Matrix Composites). A good agreement between simulated and experimental I.D. is found.

P8-8

METAL MATRIX COMPOSITES AlCu-Al<sub>2</sub>O<sub>3</sub> STUDIED BY MECHANICAL SPECTROSCOPY

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The alloy Al-4%Cu can be age-hardened and improves its mechanical properties under the effect of suitable thermal treatments. Its mechanical properties could be further increased by adding to the alloy other reinforcing phases. In this connection, metal matrix composite materials, that is Al-4%Cu reinforced with different volumetric fractions of Al<sub>2</sub>O<sub>3</sub> (Saffil) fibers have been produced by "Squeeze casting". They have been characterized by internal friction and thermo-electric power measurements, in order to identify the effect of the fibers on the precipitation of Cu, the microstructure of the alloy, and the mechanical properties.

The elastic moduli of the composites is confirmed to have better performances than the one of the not reinforced alloy, and the internal friction spectra prove to be sensitive to metal-ceramic reinforcement interactions.

In fact, after being solution-treated and quenched, the not reinforced matrix exhibits an internal friction peak P<sub>1</sub> typical of Cu in solid solution (Zener peak). After annealing some hours at 230 °C, another peak P<sub>2</sub> appears, in connection with the growth of the semicoherent  $\theta'$  precipitates in the alloy. On the contrary, after being solution-treated and quenched, the composites do not exhibit any P<sub>1</sub> peak, but a very rapid and marked appearance of the P<sub>2</sub> peak is ascertained.

Therefore,  $\theta'$  precipitation seems to be strongly accelerated in the composites, probably by the dislocations created during quenching by the thermal stresses, which originate from the different thermal expansion coefficients of the matrix and the fibers. Conclusions can be obtained about the optimum aging conditions for the composites.

P8-9  
INTERNAL FRICTION IN POLYMER NETWORKS

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Among polymeric materials, some attention has to be given to the case of polymer networks. Fully cured epoxy networks with different crosslink densities and flexibilities are studied in this papers. The crosslink density was changed by preparing epoxy networks from mixtures of difunctional and tetrafunctional amine hardeners. The flexibility of the macromolecular chains between crosslinks was varied by changing from aromatic to aliphatic epoxy prepolymers.

These polymers display several mechanical relaxation phenomena. Some of them are related to localized molecular motion (conformational changes, rotation of some molecular units,...). The main difficulties are to identify the structural units involved in these relaxations (e.g., using NMR CP-MAS) and to define the potential well determining the characteristics of the relaxations (molecular mechanics seems to be the powerful tool for that purpose).

As for other polymers, a main relaxation is observed associated to the glass transition phenomena. The theory based on the concepts of quasi-point defects and hierarchically constrained molecular dynamics is applied to the case of crosslinked polymers. The developed model previously used to explain the rheological behavior of linear amorphous polymers works successfully for the dynamic mechanical properties of these epoxy networks in their glass transition region. The analysis of molecular motions made in the small deformation mode allows us to describe the plastic behaviour of these networks in the sub-T<sub>g</sub> region.

P8-10  
DAMPING IN DISPERSION STRENGTHENED ALUMINUM ALLOYS: A MAXIMUM WITH  
HIGH ACTIVATION ENTHALPY

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Dispersion strengthened aluminum alloys are promising candidates for applications at high homologous temperatures. Besides their good creep resistance these materials exhibit a high damping capacity. The influence of various lattice defects on the damping behavior is by no means clear. To get more insight into the fundamental mechanisms the damping behavior of dispersion strengthened aluminum alloys was studied more systematically for various microstructural conditions.

An inverted torsion apparatus was applied for mechanical loss measurements by using the free decay method (1 to 10 Hz) or forced vibrations down to 10<sup>-3</sup> Hz, respectively. Four different maxima are observed between 200 K and 773 K. The maximum at T ≈ 600 K which has remarkable properties is described in detail in this work. The activation enthalpy of this maximum (H = 2.2 to 2.6 eV) which was determined from the shift of the maximum temperature with measuring frequency between 0.02 and 10 Hz is much higher than that for self diffusion in aluminum (1.48 eV). Additionally, the relaxation strength of the maximum depends on the frequency of measurement.

The high activation enthalpy can be explained by a model which considers the nucleation of jogs in climbing dislocations. In this model it is assumed that the dispersoids acts as firm pinning points for the dislocations. By taking into account different energy levels for the bowed and unbowed dislocation configurations, the dependence of the relaxation strength on the measuring frequency can be explained qualitatively.

P8-11  
THE MECHANISM OF ULTRASOUND ATTENUATION IN SINTERED METAL POWDER  
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Recently, the vibrational dynamics of a percolating network has generated explicit realization of the excitations. Such percolating networks are known to have a homogeneous (Euclidean) structure at long length scales, but have a self-similar (fractal) geometry at short length scales. The transition between these two regions occurs at the percolation correlation length  $\xi$ . Consider the scaling behavior of the density of vibrational states, write  $N(\omega) \sim A \omega^{-1} f(\omega/\omega_\infty)$ ,  $\Delta N(\omega) \sim A \omega_\infty^{-1} \Delta f$ . Crossover frequency  $\omega_\infty$  can be defined ( $\omega < \omega_\infty$ , Phonon;  $\omega > \omega_\infty$ , fracton).

The vibrational localization in sinter can be interpreted in terms of a crossover from phonons to fractons, we consider the scaling of the ultrasonic attenuation with the percolation correlation length  $\xi(f)$ . The ultrasonic wave intensity  $I(x)$  fall off as  $\log^{-1}(\omega) \sim I(x) \sim \log^{-1}(\omega)$ . At higher frequencies, there is a rapid decrease in localization length with frequency.

Williams et al have calculated phonon eigenstates for disordered systems. The density of vibrational state:

$$g(\omega) = \frac{\omega}{\pi} K \left[ \frac{\omega^2(8-\omega^2)}{16} \right],$$

where  $K$  is the complete elliptic integral of the first kind. The localization length is short (strongly localized) as the disorder is increase.

P8-12  
MECHANICAL DAMPING AND DYNAMIC MODULUS BEHAVIOR OF GRAPHITE-ALUMINIUM ALLOY COMPOSITES

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Metal-matrix composite materials have attracted great attention due to their excellent structures and properties. In recent years, however, only limited papers reported the research on the damping capacity of fiber-enhanced composites such as SiC/Al, Al/W, Gr/Al. Especially few knowledge was known about nonlinear internal friction features of composites. Therefore it is necessary to develop thorough study in this field.

In the paper, internal friction method was used to study the damping behavior and elastic modulus at low and acoustic frequency for graphite-aluminium composites with various graphite content. The intense nonlinear internal friction effects exhibited by composites were observed and the comparison with that in matrix and pure graphite were made. Internal friction spectra were measured as functions of frequency ( $5 \times 10^{-3}$ –50 Hz) and temperature. Three internal friction peaks named as 60°C, 160°C and grain boundary peak (200°C) respectively were observed and studied. The initial exploration was made on the nonlinear damping mechanism and other experimental phenomena of Gr/Al composites.

P8-13

APPLICATION OF LOW FREQUENCY INTERNAL FRICTION TECHNIQUE IN ROCKS

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Rock is a porous material which is an essential object in the research of attenuation law of earthquake. The distribution of cavity and the reserve of liquid in rocks that greatly affect the physical properties now are very attractive subjects. Since the quality factor  $Q$ , i.e., inverse of internal friction, can reflect the state of rocks, a lot of research have been made mainly by ultrasonic wave technique.

In this paper, the first investigation on granites was made by low frequency internal friction technique. Abundant internal friction phenomena and special elastic modulus behavior in wide temperature range were firstly observed and studied. It is very interesting to find the considerable amplitude-dependent effect behaved by granites. A high internal friction peak at 80°C which only appears in partially water-saturated granites was systematically studied. The other broad internal friction peak in the range of 150-300°C was also studied as due to the movement of particle boundary. A 4-parameter mechanical model describing solid-liquid complex body was firstly proposed based on the friction behavior of viscous water with boundary of cavity. Theoretical treatment gives reasonable explanation to the above two internal friction peaks.

P8-14

STUDY BY MECHANICAL SPECTROSCOPY OF THE DISLOCATION SUBSTRUCTURE IN AN ALUMINUM MATRIX COMPOSITE REINFORCED BY ALUMINA SAFFIL FIBERS

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The low frequency internal friction spectrum and elastic modulus  $G$  exhibited by aluminum matrix composites reinforced by different volumetric fractions of short, non oriented alumina fibers, have been studied in the temperature range between 100 K and 800 K.

The spectra of the composites are characterized by internal friction maxima located at roughly the same temperature ranges as those found in the Al matrix, and attributed to relaxation mechanisms involving thermally activated dislocation motion. As in monolithic aluminum, these internal friction peaks as well as the high temperature background may be correlated with the observation of different dislocation substructures. Nevertheless the addition of fibers modifies their characteristics, specially their stability against high temperature annealing.

A new feature appearing in composite spectra is an hysteretic behaviour (the spectrum during heating is different than that observed during cooling) which increases in magnitude as the content of fibers becomes higher.

Concerning the elastic modulus it has been found to grow in a linear way as the amount of fibers increases but no satisfactory accord with predictions of existent models may be established. On the other hand, modulus measurement as a function of the number of thermal cycles imposed to the composite provides information about the evolution of damage in the material.

**P8-15**

**DAMPING AND MODULUS OF EPOXY MATRIX COMPOSITE BY DISPERSION OF NANO-  
Al<sub>2</sub>O<sub>3</sub> PARTICLES**

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The dynamic mechanical properties were studied in epoxy matrix composite by dispersion of nanoscale Al<sub>2</sub>O<sub>3</sub> particles. The paper mainly focused on the influence of the embedded Al<sub>2</sub>O<sub>3</sub> particle composition on the  $\beta$ -relaxation spectrum in the epoxy, especially on the activation energy  $H$  associated with the relaxation. Finally, the interaction between the nanoscale particle and polymer was discussed.

**P8-16**

**STABLE RELAXATIONS IN MULTIPHASE Al-Zn ALLOY**

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The experiment on a forced vibration pendulum reveals the stable damping in a multiphase Al-Zn eutectoid alloy as three kinds of relaxation processes: One is linear, and two are nonlinear. The characteristics of frequency, amplitude, temperature, and process activation energy for each kind of relaxation are presented and the correlation among those relaxations are discussed as well.

# P8-17

## STUDY ON THE ULTRASONIC VELOCITY OF Cu-CARBON FIBER COMPOSITES

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The ultrasonic velocity of Cu-Carbon fiber composite was measured using the laser ultrasonics technique. A ruby laser was used as an ultrasonic source and a capacitor transducer was used as a receiver. A series composite with fixed component Cu and various component carbon fiber was used as sample. The fractions of carbon fibers for this series sample are 5, 10, 15, 20, 25 and 30 per cent respectively. The value of velocity depends on the fraction of carbon fiber, porosity and texture of composite. The values of velocity decrease with the fractions of carbon fiber components increase, when the fraction is less than 20 per cent. The rate of velocity value change tends to slow when the fraction is more than 25 per cent. The elastic constants are obtained from the values of velocities measured and the mass densities of composites. The comparison between two velocities values measured for two kinds Cu-Carbon fiber composites with difference texture, and the comparison between the measurement methods using ultrasonics and that using laser ultrasonics method are given in this paper.

# P8-18

## THE EFFECT OF STARTING TEMPERATURE ON THE INTERNAL FRICTION BEHAVIOUR OF PA1010 AND ITS BLENDS

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In the process of internal friction measurement of polyamide 1010 (PA1010) and its blends with bisphenol A polycarbonate (PC), it was found that the starting temperature for the measurement exerts remarkable influence on the relaxation features of PA1010, i.e. both the peak temperatures of  $\alpha$  relation ( $T_\alpha$ ) and  $\beta$  relaxation ( $T_\beta$ ) of PA1010 shift to higher temperatures with increasing the starting temperature for the measurement ( $T_0$ ). When  $T_0 = -160^\circ\text{C}$ , for example,  $T_\alpha = 21.3^\circ\text{C}$  and  $T_\beta = 83.4^\circ\text{C}$ , but in the case that  $T_0 = -130^\circ\text{C}$ ,  $T_\alpha = 40.6^\circ\text{C}$  and  $T_\beta = 75.9^\circ\text{C}$ . And furthermore, the half-width of  $\beta$  relaxation time distribution function in PA1010 increases with a rise in  $T_0$ .

Due to the fact that these phenomena could be observed only in crystallized polymers (such as polyphenylene sulfide, polyetheretherketone, etc.) and their blends, the relaxation characteristics of amorphous phase restrained by crystalline region in the polymer should be responsible for such an influence. The theory of polymer conformation is applied to the analysis and discussion.

P9-1  
THE ELASTICITY STUDY OF Al AND Ag NM-FILMS

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Understanding of the elastic property of metal nm-films is important for the semiconductor technology, and is also an interesting subject of materials science. Berry found that the feature and mechanism of the anelastic relaxation relevant to grain boundaries in Al 100nm-films are different from those in bulk Al. Below 100nm, in our previous work we observed that the Young's modulus,  $E$ , of Al nm-films deposited in a vacuum of  $10^{-3}$ Pa decreases with decreasing thickness,  $d$ , especially below 50nm, suggesting an increasing effect of grain boundaries at small  $d$ 's. In the present paper, we investigated the elastic properties of Al and Ag nm-films for  $d \geq 3$ nm: Al and Ag nm-films were deposited on the Si thin-reeds, here in a vacuum of  $10^{-4}$ Pa.  $E$  in the as-deposited nm-films was determined from the changes in the resonant frequency of the Si thin-reeds.

Al nm-films:  $E$  in the present nm-films is, in general, higher than that in Al nm-films of the same  $d$  used in the previous paper, suggesting that the mean planar-diameter of crystalline grains,  $R$ , in the films is larger for the nm-films deposited in the higher vacuum. Here,  $E$  shows good agreement with  $E_b$  in bulk Al for  $d > 20$ nm. With decreasing  $d$  below 20nm,  $E$  shows a strong decrease which is followed by a saturation, where  $E$  decreases to about  $E_b/3$  at  $d \approx 3$ nm. The  $E$  vs.  $d$  data can be explained by assuming that  $E$  in the sub-region of thickness  $t_{GBR}$  near grain boundaries decreases to  $E_{GBR}$ .  $E$  in the remaining remains  $E_b$ , and  $R=d$ , where the best fit gives  $t_{GBR} \approx 1$ nm and  $E_{GBR} \approx 2 \times 10^{10}$ Pa.

Ag nm-film: The as-deposited films are a polycrystalline film with the large  $R$  of about  $10\mu\text{m}$ .  $E$  shows a slight decrease with decreasing  $d$ , and the preliminary-data can be explained by assuming that  $E$  in the sub-region of thickness  $t_{SR}$  near the surface decreases to  $E_{SR}$ , where the tentative fit gives  $t_{SR} \approx 1$ nm and  $E_{SR} \approx 5 \times 10^{10}$ Pa. The internal friction reveals a very sharp peak at 155K. The 155K-peak shows no temperature-shift for varying frequency and also between heat-up and cool-down measurements. We surmise that the 155K-peak reflects some atomic process relevant to the interface between Ag and Si.

P9-2  
THE ANELASTIC STUDY OF Ag/Pd MULTILAYERS

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The observation of 200-400% increase in the biaxial elastic modulus,  $Y$ , in Au/Ni, Cu/Ni, Cu/Pd and Ag/Pd multilayer films (MLF's) near the modulation wave length,  $\lambda$ , of 2-3nm reported by Hilliard and his co-workers ignited the investigation of the so-called supermodulus effect (SME). However, many questions relevant to SME still remain unsolved, and whether SME found in early observations is intrinsic or not is in dispute. To resolve the controversy, we reexamined the elastic property of Ag/Pd MLF's by means of the advanced vibrating-reed technique.

The Ag/Pd MLF's with the fixed chemical composition of  $\text{Ag}_{50}\text{Pd}_{50}$  were deposited on the (100)-surface of Si-reeds at room temperature by the rf-sputtering method. The as-deposited Ag/Pd MLF's here were a polycrystalline MLF without crystallographic orientational-anisotropy. The Young's modulus,  $E$ , in the as-deposited Ag/Pd MLF's was determined from the changes in the resonant frequency of the Si-reeds due to the deposition.  $E$  observed in the Ag/Pd MLF's shows substantial increases in the range of  $\lambda$  between 1.5 and 5nm, two maxima beyond 200% at about 2.1 and 2.8nm and a dip at about 2.3nm. In situ heat-up measurements show the following: For the Ag/Pd MLF's which show the relatively small SME in the as-deposited state,  $E$  decreases with increasing homogenization at elevated temperatures, and after the completion of the homogenization at about 780K,  $E$  decreases to  $E_b$ , where  $E_b$  is  $E$  calculated for the homogeneous and polycrystalline  $\text{Ag}_{50}\text{Pd}_{50}$ . In contrast, the Ag/Pd MLF which shows the significant SME in the as-deposited state stands for the homogenization even at 805K, and  $E$  remains high. The former fact proves that SME observed here reflects the intrinsic elastic property of the Ag/Pd MLF's. The latter fact suggests that in the Ag/Pd MLF's showing the significant SME, the atomic diffusion is retarded, indicating that SME is associated with some change in the interatomic potential. The present results also indicate that the  $\langle 111 \rangle$  texture in Ag/Pd MLF's suggested in the early observations for  $Y$  is not a necessary condition to observe the strong SME.

**P9-3**

**THIN FILMS CHARACTERIZATION USING GENERALIZED LAMB WAVE AND HARMONIC GENERATION**

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A mechanical characterization method of a thin film on a semi-infinite substrate is proposed. We applied our acoustic nondestructive technique on a system which consists of an elastic, isotropic layer of constant thickness deposited on an elastic, isotropic substrate.

A Continuous Wave Scanning Acoustic Microscope (CW-SAM) was used to propagate surface modes in the specimen and to measure the dispersion curves. A numerical inversion procedure was achieved and tested for the dispersion relation of generalized Lamb waves (surface acoustic waves) propagating in a thin film on a substrate. This procedure allows the determination of the following parameters of the film: longitudinal elastic wave velocity, transverse elastic wave velocity, density or thickness. Possibilities and limitations of this method are studied and tested on several samples.

An application to interface characterization is suggested. By comparison of the dispersion curves shape and position with different boundary conditions (perfect and weak interface) at the interface and by observation of the harmonic generation phenomena due to the high non-linearity of weakly bonded interfaces, information on the adhesion of the film on the substrate could be obtained.

**P9-4**

**PREPARATION AND CHARACTERIZATION OF (Tb,Dy)Fe<sub>2</sub> THIN FILMS FOR A SURFACE ACOUSTIC WAVE DEVICE**

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Thin films of giant-magnetostrictive (Tb,Dy)Fe<sub>2</sub> intermetallics were prepared for the characterization of their magnetostrictions. Ion beam sputtering with a plasma filament ion source, flash evaporation and ion plating were used for film preparations. The ion beam sputtering and flash deposition yield films with little deviation in the concentration from that of source materials, while ion plating method results in very different concentrations of film and the source material. The deposition conditions give also a large difference in the magnetic properties.

**P10-1**  
**ACROBATIC BEHAVIOR OF THE FREQUENCY AND THE TEMPERATURE DEPENDENCES OF  
THE CLASSICAL SOUND ATTENUATION IN SEMIMETAL**

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The classical sound attenuation due to the carrier in semimetal has been investigated under zero magnetic field for the first time, which is a fundamental information for the transport phenomena. We measured the attenuation coefficient  $\alpha$  of the longitudinal sound waves in  $\text{Bi}_{1-x}\text{Sb}_x$  ( $x = 0, 1.0, 3.6, 8.6 \times 10^{-2}$ ) single crystals in the frequency range of  $10 \text{ MHz} \sim 300 \text{ MHz}$ , in the temperature range of  $11 \text{ K} \sim 300 \text{ K}$ . We calculated  $\alpha$  on the two band model by using the polarization function which is proportional to the classical complex conductivity including the diffusion effect. The calculated results shows a unique behavior for the frequency dependence and the temperature dependence which are very different from those in metal.

The agreement between the experimental results and the calculation is good. As the absolute value of  $\alpha$  and the curvature of the frequency dependence are very sensitive to the relaxation time of the carrier  $\tau$ , we could determine the relaxation time in dilute  $\text{Bi}_{1-x}\text{Sb}_x$  alloys by the comparison with the experimental result and the calculation with good accuracy. Consequently, its value in dilute alloys has become one or two orders shorter than that in pure Bi with  $\tau \sim 10^{-10} \text{ sec}$ , although the resistivity of the dilute alloys only differs by a factor less than 2 from that of Bi.

**P10-2**  
**COMPUTER EXPERIMENT OF PHONON TRANSPORT IN CRYSTALS WITH DEFECTS**

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Molecular dynamics computer simulation has been started to investigate transport of phonons in crystals containing point defects. One-dimensional mass-spring model crystals were used for the simulation. As an example, a random mass-defect model was adopted as the model crystal. In the model, a certain amount of different-mass defect atoms are randomly distributed among body atoms, the interatomic spacings are all the same, and the force constants between all atoms are the same. Crystal anharmonicity up to the fourth order is considered for the interatomic potential. A choice is made for the strength of anharmonicity with referring the order-of-magnitude ratios of the higher-order elastic constants of usual materials. The number of atoms in the model crystal is 550, and a line of 150-atom heat sink atoms is attached to the right end of the crystal in the phonon propagation experiment.

The crystal is firstly thermalized. Namely, an input phonon pulse is applied to an end of isolated crystal, and energy equipartition between the atoms of the crystal is realized after a prolonged time, and thus a finite crystal temperature can be set. Then the heat sink is connected, and an input phonon pulse is applied to the other end of the crystal. The input phonons propagate along the crystal and finally reach the heat sink. The heat sink can perfectly eliminate the phonons coming from the crystal. A transient propagation of phonons can thus be observed in the model crystal. The state of the propagating phonons is represented by locally averaged vibrational energies of atoms, and time variation of the energy is observed at a definite position of the crystal. A diffusive behavior of phonon transport can be seen in the crystal, and thermal diffusivity is obtained by analyzing the energy-vs-time data.

The thermal diffusivity is to be determined under various experimental conditions. The study is now being continued to investigate the dependences of diffusivity on the mass(defect)/mass(body) ratio, defect concentration, crystal temperature, and input phonon frequency.

**P10-3**  
**ULTRASONIC ATTENUATION IN SEMICONDUCTORS**  
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Grüneisen parameters, non-linearity constants and ultrasonic attenuation due to phonon-viscosity mechanism and thermoelastic loss have been evaluated in semiconducting crystals, InP, InAs and GaP at 300 K along various crystallographic directions of propagation for longitudinal and shear waves using elastic data. Ultrasonic attenuation due to thermoelastic loss has been found to be negligible in comparison to phonon-viscosity loss divulging the fact that most of the acoustical energy is either used up in equalising temperature difference among various phonon modes or producing heat. Results are in agreement with experimental results available.

**P10-4**  
**ULTRASONIC ATTENUATION STUDIES OF DIELECTRIC CRYSTALS**  
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Ultrasonic attenuation due to phonon-phonon interaction and thermoelastic loss, non-linearity constants and drag coefficients on edge and screw dislocation motions have been estimated in two dielectric crystals viz. CaO and MgO from 50 K to 500 K along  $\langle 100 \rangle$ ,  $\langle 110 \rangle$  and  $\langle 111 \rangle$  directions of propagation for longitudinal and shear waves for different polarization directions of shear waves. Using electrostatic and Born-Mayer potentials, second and third order elastic constants have been obtained at different temperatures to obtain Grüneisen parameters, hence non-linearity constants, which in turn have been used to evaluate ultrasonic attenuation. Temperature variation of the ultrasonic attenuation has been found as observed experimentally.

ULTRASONIC ATTENUATION BELOW  $T_c$  IN HEAVY-FERMION SUPERCONDUCTORSY.N. Huang, H.L. Zhou, and Y.N. WangNational Laboratory of Solid State Microstructures,  
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Up till now, in order to explain the experimental results of ultrasonic attenuation for heavy-Fermion superconductors (HFSs), various theories have been developed and cheerful progress has been made. However, the special characters of ultrasonic attenuation for HFSs indicated by experiments, which are quite different from those of conventional superconductors, can not be interpreted simultaneously. With taking account of quasi-particle recombination due to strong-coupled correlation in heavy-Fermion superconductors, we get satisfactory results. The calculated ultrasonic attenuation below  $T_c$  indicates that: (i) there appears a  $\lambda$ -shaped peak near below  $T_c$ , with the peak temperature  $T_p$  independent of frequency and the peak height  $\alpha_s(T_p)/\alpha_n$  decreasing slowly while increasing frequency ;and (ii) there is a power-law of attenuation  $\alpha_s(T)/\alpha_n$  at lower temperature. These results are fairly in agreement with the available experimental data so far.

**P11-1**  
**GRAIN BOUNDARY RELAXATION IN PURE METALS WITH FCC STRUCTURE**

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Low frequency internal friction spectrum of polycrystalline FCC pure metals with the different stacking fault energy (Ag, Cu, Ni) possesses with three maxima. These maxima exist in annealed metals and their intensity and activation energy depend on the structure of the specimen. Structure investigations allow to bound every maximum with definite kind of grain boundaries: general kind, special and 'bamboo'.

**P11-2**  
**PLASTICITY OF PERFECT CRYSTALS**

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We investigate the behavior of complex defects in the crystal in the parameter region, where the rotation relaxation is very quick. The defects could be the small dislocation loops, vacancies (or interstitials) complexes. In this case it is possible to derive a system of hydrodynamic equations, describing bulk crystal dynamics, and a set of boundary conditions for both crystal-crystal and crystal-liquid interfaces.

A certain limit of boundary conditions reproduces the well known result for vacancy concentration near the boundary, introduced by Herring.

Obtained system describes an interesting phenomena of "equilibrium plasticity" which results in the plastic deformation (the change of crystal shape which do not correspond to the change of the crystal elementary cell shape), that under certain conditions is reversible i.e. vanishes after the removal of the external load.

The obtained equations can be used to describe acoustic waves propagation and other long wavelength vibrations in the crystals with complex defects.

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P11-3  
VARIATION OF THE GRAIN BOUNDARY RELAXATION STRENGTH AND THE GRAIN  
BOUNDARY MODEL OF DISORDERED ATOM GROUPS

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According to the grain boundary model of disordered atom groups, the relaxation strength of grain boundary relaxation varies with the number of disordered groups existing in the grain boundary and the degree of disorderness of each group. It was found recently that the relaxation strength of grain boundary in polycrystalline and bamboo crystalline aluminium drops with decreasing temperatures and reaches an undetectable value at a temperature  $T_0$  which is about  $1/2T_m$  where  $T_m$  is the melting temperature of bulk specimen. The existence of such a temperature  $T_0$  was confirmed lately by measurements of micro-creep and internal friction (in forced vibration) for aluminium bicrystals with random boundaries and special boundaries (with a definite  $\Sigma$  value). These results are in conformity with those obtained by molecular dynamics simulation.

Basing on the results of anelastic measurements and MD simulation, we consider that in the case of special boundaries, the temperature  $T_0$  reflects a pronounced transition of the grain boundary from the CSL structure to that of disordered atom groups. However, the situation is different in the case of random boundaries which may consist of a mixture of disordered structure and CSL structure. The variation of the number and the disorderness of the disordered group at  $T_0$  should be smoothly for the disordered component of the random boundary and the occurrence of a weakly detectable transition at  $T_0$  may be considered to be due to the abrupt transition of the CSL component of the random boundary.

P11-4  
RELATION BETWEEN FABRICATION PARAMETERS AND GRAIN BOUNDARY INTERNAL  
FRICTION PEAK IN COMMERCIAL Al-Mg-Si ALLOYS

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The polycrystalline Al-Mg-Si alloys (6063) have an internal friction peak at 480K (1Hz) which was attributed by S.E.Urreta de Pereyra et al.(1989) to a grain boundary sliding in the presence of particles by analogy with the pure Al or Al-Si grain boundary peak.

The aim of this work is to check the influence of certain fabrication parameters (alloy chemical composition, extrusion temperature and the homogenization conditions) on the grain boundary internal friction peaks of a lot of alloys.

Grain size determinations on the internal friction samples were made following the method indicated in the 112-74 ASTM Standard method, in order to make a correction on the internal friction maximae, previously background subtraction. This allows us to compare results on samples of different grain size (between 34 and 57 $\mu$ m).

The main result is that the corrected internal friction peak shows an increasing dependence with the Si composition of the studied alloys. The extrusion temperature is the parameter that indicates a separation the alloys into different two groups. The homogenization parameters seems to be not important in this case.

It is noticeable the fact that we do not observe the dependence on the excess Si (respect to the stoichiometric chemical composition) predicted by others authors, but a dependence with the Si content.

**P11-5**  
**INVESTIGATION OF GRAIN BOUNDARY SEGREGATION IN Cu(Sb) AND Cu (Pb) SYSTEMS  
BY INTERNAL FRICTION**

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Since the first famous study of Kê made on polycrystalline material in order to investigate the effect of grain boundary on internal friction there has been a long discussion about the origin of the grain boundary peak.

The aim of the present study was to investigate the influence of grain boundary impurities on the internal friction spectra, mainly on the grain boundary peak.

Our measurements were carried out in a torsion pendulum in the temperature range of 300-700 K. The grain structure of 4N copper wire was stabilized by annealing at 725 K and by internal friction measurement of pure Cu. After stabilization a Sb or Pb layer of 0.1 micrometer was evaporated on the wire. The impurity atoms (Sb, Pb) were introduced into the grain boundaries by a controlled grain boundary diffusion (mainly in C-kinetics). The grain boundaries of copper wire were saturated with Pb or Sb atoms with this method.

Our results are as follows:

- the level of both low- and high-temperature background increased,
- the Sb atoms did not influenced the pure copper grain boundary peak, if the measurements were carried out at 10 Pa,
- a new peak appeared at about 470 K on the spectra of Cu(Sb) system measured at  $5 \cdot 10^2$  Pa.
- the Pb atoms changed the internal friction spectra of pure copper:
  - a) a new peak appeared at about 323 K (it is close to the temperature of grain boundary peak of pure lead),
  - b) the height of the copper grain boundary peak decreased.

**P11-6**  
**ON THE ASSOCIATION OF THE GRAIN BOUNDARY KÊ PEAK WITH THE STRUCTURE OF  
THE LARGE ANGLE GRAIN BOUNDARIES**

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Inelastic response of the large angle grain boundary ensembles was studied within the framework of the classic Kê model. It was shown, that each boundary of the ensemble at some temperature  $T_s$  undergoes structure transformation and essentially change response on the external disturbance. Expression for the part of sliding boundaries as a function of homological temperature was obtained for the simple statistic models of the boundary ensemble and for a set of simplifying assumptions on the angle and temperature dependence of the surface tension of the large angle boundaries. It was shown that expression  $T_s \approx (0.3 \div 0.4)T_m$ , where  $T_m$  is melting temperature, is valid for many pure metals. Satisfactory coincidence with the experimental results was obtained for the dependence of the strength of relaxation for Kê peak on its homological temperature for the set of pure metals.

**P11-7**  
**INTERNAL FRICTION OF GRAIN BOUNDARY OF SEVERAL IRON BASE MATERIALS**

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To further demonstrate the nature of  $K_\alpha$  peak and probe into its mechanism, a method distinguishing grain boundary peak by doping with surface active elements was developed, and the grain boundary internal friction (GBIF) of the high pure Fe, Fe-P, Fe-P-La and Fe-C-Nb alloys were studied. In view of such experimental facts that the  $K_\alpha$  peaks in the alloys of Fe-P-La and Fe-C-Nb evidently reduced, and almost eliminated, until were completely inhibited as the compounds containing La, P, and/or Nb etc produced, increased and became a net along grain boundaries and so on, it followed that the  $K_\alpha$  peaks in the tested alloys were all GBIF peaks. It was further pointed out that GBIF is induced by the movements of both the defects with atomic dimension, namely point defects, in grain boundaries and/or their clusters under stress, and the movements of the GBIF sources perform by means of atomic diffusion. The GBIF sources are a region of high energy, and are readily occupied by foreign atoms, which even result in the segregation of the foreign atoms, the formation of both the clusters and precipitates of second phase. The latter processes are responsible for the phenomena that the PM peak, the SS peak, the SS of cluster occurs, and the GBIF peaks lower and even eliminate completely.

**P11-8**  
**RESONANCE ULTRASONIC ATTENUATION BY THE IMPURITY LAYER ADSORBED ON INTERFACE**

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A total set of dynamical boundary conditions for the elastic wave on interfaces or planar fault stackings in crystals are derived. A new mechanical variable, the elastic displacement of two-dimensional adsorbed impurity layer, is introduced. This variable describes the internal dynamical degree of freedom, associated with an additional self-frequency. The existence of this degree of freedom leads to the resonance attenuation of the incident acoustic wave. The possibility of a total resonance transmission of the acoustic waves through the interface is found and the conditions of this phenomenon are studied.

**P11-9**  
**DAMPING CAPACITY OF Zn-Al ALLOY SHEETS**

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The effect of cold rolling on the logarithmic decrement  $\delta$  of Zn-Al / Zn-Al-0.02% Mg alloy containing Al less than 22% were investigated for water quenched from 573 K for 2 / 48 hours, as-rolled and water quenched from 573 K for 2 / 48 hours after cold-rolled alloy sheets. With increase in rolling reduction, the value  $\delta$  was increased. As a results of tensile properties and hardness test, elongation was increased and hardness was decreased with increase in rolling reduction for as-rolled alloys.

An increase in the value  $\delta$  was significant great for the as-rolled eutectic alloys with an accompanying work-softening effect resulted from grain refinement of lamellar structure to an equiaxed fine  $\alpha+\beta$  structure. For the water quenched after cold-rolled hyper-eutectic alloys, hardness showed lower value and indicated no significant change by varying rolling reduction.

For the microstructure of the alloys, the grains have retained equiaxed features after large rolling reduction. Further microstructural study revealed that the grain feature and structure of the eutectic alloy after 85% reduction in area was similar to those of the as-quenched alloys. The value  $\delta$  of cold-rolled alloys showed pretty close to that of the as-quenched alloys. No distinguishing change was observed between the alloys subjected to heat treatment for 2 and 48 hours after cold-rolled.

**P11-10**  
**Q-1 STUDY ON SHORT RANGE Cr DISTRIBUTION IN MARTENSITIC STEELS**

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With 11%Cr steels of MANET type, cooling with different rates from the austenitisation temperature leads to different martensite structure evolutions during tempering which are accompanied by different fracture behaviour, in particular in the ductile to brittle transition.

Data obtained from X-ray diffraction analyses, microanalysis in SEM and from the mechanical tests are accompanied by analyses of the dynamic modulus and of the internal friction spectra vs. temperature, followed after various tempering treatments, isochronal at different temperatures and isothermal for different times.

The results are consistent with the presence in martensite of C-Cr associates, deriving from previous associates present in the austenite before quenching.

The observations reported regard the behaviour after usual treatment of cooling from the austenitisation temperature, with moderate cooling rate, in comparison with the behaviour after fast cooling.

After cooling with the lower rate the evolution of the Q-1 spectra during tempering at relatively low temperatures is related to the processes through which the C atoms pass from associates to carbides in lath and PAG boundaries. At relatively high temperatures sufficient for Cr diffusion, the Cr clusters remained without C are submitted first to a process of redistribution with homogeneization; subsequently Cr enrichment near to the grain boundaries occurs, most probably connected with C escape from the grain boundaries themselves.

After fast cooling the structural characteristics giving rise to the internal friction peaks as well as to specific broadening of the XRD lines, present a greater stability vs. tempering, in the whole range of tempering temperatures taken into account. This greater stability corresponds to smaller effects of the tempering temperature on the ductile to brittle transition temperature.

According to the C-nCr associate model in question these cooling rate effects are related to the formation at high temperatures of associates having different characteristics, with specific influences on the nucleation and on the subsequent development of martensite

**P11-11**  
**STUDY OF THE HIGH TEMPERATURE INTERNAL FRICTION IN ULTRA PURE ALUMINIUM  
BY ISOTHERMAL MECHANICAL SPECTROMETRY**

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Anisothermal internal friction spectra obtained with high purity aluminium (99,9999 %) samples exhibit two relaxation peaks situated between 250 K and 550 K. The peak height and temperature were found sensitive to the vibration amplitude.

Because the internal friction in high purity aluminium is very sensitive to the temperature rate  $\dot{T}$ , isothermal experiments were carried out in a large frequency ( $10^{-4}$  Hz - 160 Hz) and vibration amplitude ( $5 \cdot 10^{-7}$  -  $10^{-5}$ ) ranges. The peak frequency at fixed temperature depends on the vibration amplitude  $\epsilon_M$ , confirming previous results but the relaxation parameters are quite different. For instance the apparent activation energy was found close to 2 eV and slightly dependent on  $\epsilon_M$ .

**P11-12**  
**TWO DIMENSIONAL MODEL OF THE INHOMOGENEOUS GLIDE OF GRAIN BOUNDARIES**

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The equilibrium equation of the applied stress, viscous glide stress and the long range stress associated with the inhomogeneous glide, was solved by using a two-dimensional model of continuous distribution of dislocations. The glide, internal friction and modulus defect were obtained as a function of the frequencies and temperatures. In order to compare the calculated results with the experimental results of the internal friction peaks observed from pure and less pure polycrystals, dicrystal specimens and the specimens with bamboo grain boundaries, the applied stress and viscosity were selected to be homogeneous or inhomogeneous accordingly. Satisfying results were obtained by the reasonable selection of viscous coefficients.

It is difficult to distinguish between the mobility of the grain boundaries and the climb of the dislocation theoretically. The thickness of the layer of grain boundaries associated with the glide process is insensitive to the results. It is suggested that a new microcreep experimental technique using a marked dicrystal in a tunnel microscope, to measure the microcreep of the markers on the surface in atomic level would be a powerful tool to study the dynamics of the grain boundaries. An international cooperation is welcome.

**P11-13**  
**A COMPREHENSIVE STUDY ON GRAIN BOUNDARY RELAXATION**  
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Anelasticity was chosen by Zener to denote that property of solids in virtue of which stress and strain are not single-valued functions of one another in that low stress range in which no permanent set occurred and in which the relation of stress to strain is still linear. Started from the Boltzmann superposition principle, the relationships between various anelastic effects (dynamic and quasi-static) derived by Zener was confirmed by Kê in his study on the grain boundary relaxation in polycrystalline specimens. However, the Arrhenius equation was assumed to be valid in grain boundary process in Kê's experiments and the internal friction and dynamic modulus were measured as functions of temperature so that a grain boundary peak and modulus defect versus temperature were observed. Whereas the Debye peak is referred to  $\omega\tau$  where  $\omega$  is the angular frequency and  $\tau$  is the relaxation time, it seems that, a precise approach is to measure the internal friction and dynamic modulus as function of frequency. Especially that the grain boundary relaxation has been found recently to exhibit nonlinear behavior in polycrystalline, bamboo crystalline and bicrystal specimens, so that a close examination on the relationships between various "anelastic" effects (linear and nonlinear) is pertinent.

This paper reports the experimental results of measurement of grain boundary relaxation versus various parameters so that the frequency curve, the temperature curve and the data of quasi-static measurement were obtained with the same specimen polycrystalline, bamboo crystalline and bicrystal specimens were used. The results were analyzed collectively and the mechanism of linear and nonlinear grain boundary relaxation was suggested conforming with the results. The mechanical model as well as the electrical model were used in the analysis and simulations.

**P12-1**  
**THE ANELASTIC PROPERTIES OF THE EARTH'S INTERIOR**

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The distribution of the elastic parameters and of the density in the Earth are the best known properties of the interior of our planet. On the contrary the anelastic and electromagnetic properties, at the best, may be defined "mysterious". In fact for long time the models of these properties were not known. The difficulties to solve the problem are: the impossibility to observe directly at a reasonable depth, the time required by the conventional laboratory experiments, the existence of a very limited number of anelastic and electromagnetic phenomena observable on the surface of the Earth with the required time length, accuracy, resolution. In recent times however more advanced techniques of laboratory experimentation and better theoretical models have been introduced which represent well, by means of derivatives of fractional order, the anelastic properties of many materials ranging from metals to composites and polymers (Bagley and Torvik 1983a, 1983b, 1986). The tests of the different theoretical models to represent the phenomenology of the Earth show that the stress strain relations with derivatives of fractional order give the best approximation of the observed anelastic phenomena (Froning and Muller 1979). It is also shown that the electromagnetic properties depending on the dielectric constant, for the uppermost layers of the Earth, are well represented by a linear relation with derivatives of fractional order.

**P12-2**  
**STRENGTH OF RELAXATION AND DISTRIBUTION FUNCTION OF AN INTERNAL FRICTION PEAK**

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A procedure, based on the mechanical properties of a *modified anelastic element* (M.A.E.) has already been developed to get a functional dependence of the real and imaginary components of the dynamical modulus or compliance. The M.A.E. is essentially a standard anelastic element except for its characteristic time that depends on frequency. The analysis of this dependence provides an analytical description of not only the dynamical properties but the distribution function, as well.

In this work it is shown that the procedure can be extended to internal friction peaks, getting not only the parameters of the distribution function but the strength of relaxation, as well. Then, this procedure is applied to different materials and the results are discussed considering several well-known distribution functions.

**P12-3**  
**HIGH TEMPERATURE BACKGROUND DAMPING**

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The high temperature background damping of several metals and alloys is analyzed, in terms of the different constitutive equations proposed in the literature for its descriptions. It is shown how, by means of a new measuring technique, it is possible, not only to obtain information on the temperature dependence of this damping, but also on its frequency dependence. In fact, not only the temperature dependence of the damping is obtained but also the derivatives with respect to the temperature and the frequency.

The procedure is applied to several hexagonal and cubic metals and alloys and the meaning of the different physical parameters obtained from the experimental data is discussed.

**P12-4**  
**ZENER RELAXATION IN BCC AND FCC ALLOYS**

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The theory of Welch and Le Claire for the relaxation strength of shear modulus in f.c.c. alloys is extended to the case of longitudinal excitations, where the relaxation of Young's modulus is involved. Not only f.c.c. but also b.c.c. lattices will be considered to obtain a complete set of equations to be used to describe the Zener relaxation strength of the torsion, Young's and bulk moduli in substitutional alloys of cubic structure.

With a single set of parameters, the equations developed appears capable of giving a satisfactory account of all properties of the relaxation strength in both alloys systems. Namely, the anisotropy of the relaxation strength and its temperature dependence, and the concentration dependence of the anelastic critical temperature.

**P12-5**  
**FRACTIONAL RELAXATION AND FRACTIONAL DIFFUSION IN ANELASTIC SOLIDS**  
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Relaxation and diffusion processes are very common and are found in many areas of physics. In particular, these processes play a fundamental role in the physical interpretation of anelasticity in solids. From the mathematical point of view they are governed, in the simplest cases, by well known linear differential equations.

The purpose of this note is to consider, after some mathematics, the physical aspects of the above processes when the first time derivative in the corresponding differential equations is replaced by a time derivative of fractional order  $\alpha$  ( $0 < \alpha \leq 1$ ). The new equations read

$$\text{Relaxation} \quad \frac{d^\alpha u}{dt^\alpha} + \frac{1}{\tau^\alpha} u = 0, \quad u = u(t), \quad t \geq 0, \quad (1)$$

where  $\tau > 0$  is a generalized relaxation time, and

$$\text{Diffusion} \quad \frac{\partial^\alpha u}{\partial t^\alpha} = D_\alpha \frac{\partial^2 u}{\partial x^2}, \quad u(x, t), \quad t \geq 0 \quad a \leq x \leq b, \quad (2)$$

where  $D_\alpha > 0$  is a generalized diffusivity constant, and  $a \geq -\infty$ ,  $b \leq +\infty$ .

Extending the classical argument of Zener, we show that our generalized relaxation process allows the thermoelastic coupling to change a purely elastic solid into a particular viscoelastic one. The new model is governed by an integro-differential stress-strain equation which generalizes the well known differential equation of Zener (standard linear solid). The relaxation function is no longer exponential in that it is represented by a Mittag-Leffler function which decays faster for short times and slower for long times. The resulting function of internal friction versus frequency is broader than Debye one, reducing to it when  $\alpha = 1$ .

As far as the generalized diffusion process is concerned, we show that equation (2) governs the propagation of stress waves in anelastic solids, which exhibit a power law creep. We present the fundamental solution, the so called Green function, in a closed form by introducing generalized Airy integrals.

**P12-6**  
**THE RELAXATION STRENGTH AND THE RELAXATION TIME IN ANELASTIC**  
**MEASUREMENTS ON FOIL-SHAPED SAMPLES**

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The relaxation strength measured in anelastic bending experiments on foils or thin plates (length, width  $\gg$  thickness) can substantially differ from the standard expression applicable in the case of rods. The effect was theoretically and experimentally studied for a special sample geometry in the case of the Gorsky relaxation [1, 2]. We present a more general classification of the torques and elastic boundary conditions in the case of bending experiments on foil-shaped samples. Using this classification scheme, we demonstrate how the relaxation strength depends on the geometry and the fixing of the sample. For two limiting geometrical situations (length  $\gg$  width and length  $\ll$  width), we present explicit expressions for the relaxation strength of foil-shaped samples, valid for cubic crystal symmetry and arbitrary crystal orientation. For Gorsky effect measurements, we derive also an expression for the effective diffusion coefficient which defines the relaxation time.

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P12-7  
ON THE RELATIONSHIP BETWEEN AMPLITUDE-DEPENDENT INTERNAL FRICTION AND  
THE YOUNG MODULUS DEFECT

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The breakaway model of amplitude-dependent internal friction (ADIF) [1] predicts the value of the ratio  $r$  of amplitude-dependent decrement to Young's modulus defect (YMD) of the order of unity and can not explain the variety of available data on  $r$ .

The analysis of the experimental data in [2] lead to conclusion, that friction theories rather than breakaway ones can elucidate the  $r$  behaviour for different materials. It was assumed, that  $r$  value reflects the difference in mechanisms, controlling the motion of dislocations in lattice.

On the basis of experimental study of reversible dislocation strain in crystals, phenomenological ADIF model of unlocalized friction type, taking into account dislocations overcoming simultaneously short- and long-range obstacles was proposed. The interconnection between amplitude dependence of ADIF and YMD and  $r$  value was obtained:  $r = 4n/(n+2)$ , that gives  $0 < r < 4$  for different values of exponent  $n$  in amplitude dependence of decrement [3]. This range in a framework of unique model covers all  $r$  values, obtained in [2] for different mechanisms of dislocations motion. It has been shown that this result is a consequence of description [4] of microplastic strain as a power law of applied stress. The experimental data available for different techniques and frequency ranges has been shown to correspond in many cases to the proposed description and indicates its validity.

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**P13-1**  
**INFLUENCE OF MAGNETIC FIELD ON INTERNAL FRICTION OF Ni**  
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Amplitude dependence of internal friction due to magnetomechanical hysteresis in pure Ni sample was tested using middle frequency torsional pendulum ( $f = 80 \text{ Hz}$ ) at room temperature. Shear strain amplitude of vibration  $\gamma$  was within the range from  $0.1 \cdot 10^{-4}$  up to  $10 \cdot 10^{-4}$ . The influence of DC magnetic field  $H_D$  and AC magnetic field  $H_A$  - synchronous with applied shear stress strain, on the magnetomechanical damping have been studied to confirm the validity of the Degauque's model of magnetomechanical hysteresis. Amplitude dependence and field dependence of magnetomechanical damping have been measured and approximated by two phenomenological formulae  $Q^{-1}(H_D, \gamma)$  and  $Q^{-1}(H_A, \gamma)$  with only four common parameters to be fitted, which are closely related to the microscopic parameters:  $\lambda_s$  - magnetostriction,  $\sigma_i$  - internal stress and to the macroscopic parameters:  $G$  - shear modulus and  $B_s$  - magnetic induction.

It was revealed that proposed formula fits very well the experimental results of the magnetomechanical damping measurements in Ni sample.

**P13-2**  
**ULTRASONIC INVESTIGATION OF  $\text{Nd}_2\text{Cu}_{1-x}\text{Zn}_x\text{O}_4$  MAGNETOELASTIC INTERACTION IN THE ANTIFERROMAGNETIC AND PARAMAGNETIC STATE**

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Ultrasonic attenuation and velocity measurements have been performed on pure and Zn-doped  $\text{Nd}_2\text{CuO}_4$  over the temperature range 140-300 K with a pulse echo overlap technique operating at 10 Mhz.

Polycrystalline specimens in form of cylindrical samples, prepared by solid state reaction were employed. The Zn doping covers the range 0-5 %.

An attenuation peak and a change of slope in the longitudinal mode velocity trend, have been observed on the pure compound near the reported Neel temperature ( $T_N$ ). The approach is similar to the one of Melcher et al. in  $\text{RbMnF}_3$ . The results indicate that the onset of a long range magnetic order at  $T_N$  affects the elastic modes.

The shift at lower temperatures of the attenuation peak observed in the Zn doped compounds, is consistent with a shift of  $T_N$  following substitution of  $\text{Cu}^{2+}$  by diamagnetic Zn, as observed in other cuprates.

**P13-3**  
**MAGNETIC PERMEABILITY DISACCOMMODATION IN  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  ALLOY**

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Recently it was found [1,2] that addition Cu and Nb to Fe-Si-B amorphous alloys allows to obtain very soft magnetic materials after their crystallization.

The aim of this paper is to study the influence of annealing on the magnetic properties of  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  ribbons. The permeability, its disaccommodation and coercivity were measured for samples in as-quenched state and after annealing at 700 K, 820 K and 850 K in vacuum of 0.01 Pa.

It was stated that the initial magnetic permeability distinctly increases after annealing at 820 and 850 K for 0.5 h and depend on the temperature of measurements.

The form of the  $\Delta(1/\chi) = 1/\chi_2 - 1/\chi_1 = f(H)$  ( $1/\chi_1$  and  $1/\chi_2$  are the reciprocal susceptibilities at times  $t_1$  and  $t_2$  after demagnetization and  $H$  is the intensity of the magnetizing field) curves showing marked maxima indicates that in the samples in as-quenched state and annealed at 700 K the disaccommodation process is primarily associated with migrational relaxations in the  $180^\circ$  domain walls. After annealing the samples at 820 and 850 K the intensity of disaccommodation drastically decreases.

The results of the isochronal measurements indicate that in the amorphous sample occurs a wide band of MPD (200-400 K) with a maximum at  $T_p = 390$  K. This band is of compound nature and two superimposing areas can be distinguished. Annealing the samples at 700 K causes the decrease of the disaccommodation intensity. The annealing at 820 and 850 K causes the drop of the MPD intensity almost to zero. It seems that this decrease of disaccommodation is connected with microstructure of these alloys and averaging out the magnetocrystalline anisotropy.

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**P13-4**  
**EVOLUTION OF MAGNETOMECHANICAL DAMPING AND MAGNETIC PROPERTIES OF  
PURE IRON SILICONIZED UP TO 6.5 % BY CHEMICAL VAPOUR DEPOSITION**

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Silicon steel is particularly suitable for transformers and motor cores, because it has good soft magnetic properties. In particular with 6.5 % Si, this material has improved performances because of the higher resistivity, lower magnetostriction and decreased magnetic anisotropy resulting from an increased silicon content. However, above 3.5 % Si, a lowered ductility makes conventional processing into textured strips extremely difficult. Another possible method is the siliconization of iron by the chemical vapour deposition (C.V.D.) method.

In this investigation we will examine the magnetomechanical damping evolution (measured at 0.7 Hz with torsional pendulum) of iron strips which have been siliconized at high temperature between 0.5 and 6.5 % (weight). Results show a strong decrease of the magnetomechanical hysteresis losses with the increase of silicon content, which is subsequent to a lowering of the magnetic core losses measured at high frequencies (over 400 Hz). C.V.D. strips are compared with conventional 3 % Si steel sheets : for 6 % Si content, the magnetomechanical and magnetic core losses are less than half of that of non-oriented Si steel, and nearly the same as that of grain-oriented Si steel.

### P13-5

## A MAGNETIC INTERNAL FRICTION PEAK IN RARE EARTH-IRON ALLOYS

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The coupling between elastic and magnetic phenomena in ferromagnetic materials often gives rise to internal friction. In the present experiment, Specimen of Rare Earth-iron alloys was made to vibrate in torsion under an alternating magnetic field. A pronounced internal friction peak was observed at 2-4 Oe in the field dependence of the internal friction. The height of the peak was raised with the increase of vibrating frequency and also changed with the composition. The highest peak was observed in the sample with the Rare Earth composition of 0.017 wt%. This kind of the peak did not appear under the same conditions with exception of using a static magnetic field.

The phenomena above can be explained by the theory of reversible and irreversible displacement of the domain boundary, proposed by T.S.Ke in 1955.

### P13-6

## MAGNETO-ELASTIC EFFECTS IN $\text{Nd}_2\text{CuO}_4$

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According to modern representations, noncollinear orthogonal antiferromagnetic structure is realized below 30 K in  $\text{Nd}_2\text{CuO}_4$  - the parent compound of high- $T_c$  superconductors with the electron type conductivity. This structure can transform into nonorthogonal non-collinear or collinear antiferromagnetic configuration. The transition into the nonorthogonal noncollinear antiferromagnetic phase in terms of symmetry is intrinsic ferroelastic one, which leads up to appearance of the orthorhombic Uxy deformation and results in substantial softening of the C66 elastic modulus. We observe such peculiarity ( $\Delta C_{66}/C_{66} \approx 5\%$ ) in a temperature dependence of the modulus near  $T = 5$  K, whereas anomalies of the C11 and C44 elastic moduli are relatively weak. A phase transition between the nonorthogonal non-collinear and collinear states is not ferroelastic one, however, it is possible to expect significant jump like C66 alterations due to exchange magnetostriction renormalization of this modulus. Such peculiarities are not observed at zero magnetic field, while field dependences of C66 demonstrate them for field directed along the (ab) plane.

Using the data obtained, the low temperature parts of the phase  $H - T$  ( $H \parallel [100]$ ,  $H \parallel [110]$ ) diagrams have been plotted. We have also studied an influence of slight, Ce<sup>3+</sup> ions substitution for Nd<sup>3+</sup> ions on the sound peculiarities character and view of the  $H - T$  diagrams. It has been discovered that Ce-doping results in significant decreasing of the ultrasonic anomalies and in suppressing of the some low temperature phase transitions.

P13-7  
RESONANT SPIN-PHONON COUPLING IN MnZn-FERRITES

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In spite of the relatively weak magnetostriction in MnZn-ferrites, the ultrasonic attenuation is predominantly determined by the coupling of phonons to the spin system. This coupling has been investigated as a function of temperature, dc-bias field and frequency for various ceramic samples of MnZn-ferrites. As a result it is shown that the attenuation is drastically reduced when the spin system is frozen by a dc-magnetic field. Variation of temperature leads to a peak in the attenuation which corresponds to the peak in the imaginary part of the susceptibility, as was confirmed by an independent measurement. The results can be satisfactorily interpreted in terms of a simple model.

P13-8  
"GIANT"  $\Delta E$ -EFFECT IN AMORPHOUS ALLOYS WITH THE PERPENDICULAR MAGNETIC ANISOTROPY

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The important peculiarity of the "giant"  $\Delta E$ -effect observed in amorphous ferromagnetic ribbons with transverse magnetic anisotropy induced by thermomagnetic or thermomechanical treatment is a strongly nonmonotonous dependence vs magnetic field. We observed in amorphous ferromagnetic Fe-B-P alloys an analogous nonmonotonous  $\Delta E$ -effect dependence resulting from the heat treatment at the temperatures above 300°C, but without magnetic field or mechanical stress. The analysis of the orientational dependences of the  $\Delta E$ -effect and dynamic magnetization curves leads us to the conclusion that under heat treatment of amorphous Fe-B-P ribbons the perpendicular (with easy axis being perpendicular to ribbon plane) magnetic anisotropy is induced. We suppose that the induced perpendicular magnetic anisotropy is caused due to change of the chemical composition of the nearsurface layer under heat treatment. This thermally induced perpendicular anisotropy and attributed  $\Delta E$ -effect are steady in time and remain usually unchanged after removing (by chemical or ionic etching) the nearsurface layer. However, if the depth of the removed layer is greater than certain value, the  $\Delta E$ -effect and dynamic magnetization curve become instable in time. They remain the same immediately after etching but then relax with time to the state peculiar to the samples without induced anisotropy. The characteristic time of this change is about some hours. The previous state can be restored by repeated heat treatment or by additional etching, but in the latter case the  $\Delta E$ -effect and magnetization curve are again obtained unstable and relax in time. The possible causes of such a behavior are discussed.

**P13-9**

**EFFECT OF MAGNETIC FIELD ON THE VELOCITY OF SURFACE ACOUSTIC WAVES ON THIN FILMS OF GIANT MAGNETOSTRICTIVE ALLOYS**

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The wave velocity of a surface acoustic wave (SAW) on giant magnetostrictive (Tb,Dy)Fe<sub>2</sub>-thin films was measured. This velocity depends on the mechanical constants of the material which can be changed by magnetostriction under an applied magnetic field. The large magnetostrictions of the (Tb,Dy)Fe<sub>2</sub> materials, which are 10 to 100 times larger than those of other magnetostrictive metals like Ni, can therefore be used for SAW devices such as delay lines, signal converters and -filters, which are then tunable by external magnetic fields. Thin films of magnetostrictive materials were produced by sputtering on piezoelectric LiNbO<sub>3</sub> substrates, equipped with SAW-exciting interdigital electrodes. The influence of film preparation, film thickness and magnetic field was investigated.

**P13-10**

**FIELD AND TEMPERATURE ANOMALIES OF THE ULTRASONIC ATTENUATION IN Ni AND Co SINGLE CRYSTALS**

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It have been several different models proposed, explaining field and temperature anomalies of the ultrasonic attenuation in Ni and Co single crystals [1,2,3] Predictions of traditional theory is thought by some authors [2] inconsistent with experiment. We analyzed published experimental data along with our own recent measurements and came to the conclusion, that neither of the above mentioned models is satisfying, but more objective insight on the nature of the phenomenon may be achieved, taking into account reasonable propositions from [1,2,3] and some factors, which influence upon the magnetoacoustic interaction. We proved, that under realistic conditions theory [4] is capable to predict at least qualitatively the attenuation anomalies.

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## INTERNAL FRICTION STUDY OF DOMAIN WALL MOTION IN METALLIC GLASS FeSiB

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The internal friction (IF) measurement is used to study the domain wall motion in metallic glass FeSiB. The IF and modulus  $(f f_0)^2$  samples undergoing a uniform magnetic field sweep is measured with a mini-inverted vacuum torsion pendulum in the field range 0-8 Oe. The field sweeping increases the IF and the modulus defect and the characteristic quantity is the difference between the IF measured in the field sweeping and that in a static field, which is called dynamic IF  $Q_{dyn}^{-1}$ . The  $Q_{dyn}^{-1}$  shows a viscoelastic feature; it increases with increasing  $(H/\omega)^n$  where  $H$  is the field sweep rate  $\omega$  the angular frequency of the alternating strain. The static IF shows a frequency dependence as  $Q_s \propto \omega^m$ . For FeSiB metallic glass  $n=0.66$  and  $m=0.6$ . The behavior of dynamic IF shows that it is caused by the domain wall motion in field sweeping and thus is related to a magnetomechanical interaction. The coupling coefficient  $\alpha$  of domain wall with oscillating force is also obtained as  $\alpha(\omega) \propto \omega^{-1}$  and  $l=0.19$ .

A magnetomechanical interaction is proposed to explain the dynamic IF and static IF, and the unique behavior that dynamic IF increases with  $(H/\omega)^n$  and static IF increases with  $\omega^m$ . A phenomenological model is proposed and a two-term parametrization expressions are used to fit experimental results.

**P14-1**  
**INTERNAL-FRICTION "TENSOR" OF A COPPER MONOCRYSTAL**

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We applied ultrasonic-resonance spectroscopy to a copper monocrystal to determine both the real and imaginary parts of the elastic stiffness  $\tilde{C}_{ij} = C_{ij} + iC_{ij}^*$ . Resonance frequencies give the usual Voigt elastic stiffnesses  $C_{ij}$ . Frequencies involved are high kilohertz and low megahertz. Resonance peak widths give the complementary internal-friction components  $C_{ij}^*$ . We attribute the  $C_{ij}^*$  to dislocations, and we interpret our results using a Koehler-Granato-Lücke vibrating-string model. As expected,  $(1/3)(C_{11} + 2C_{12})^*$  is small. In (111)-glide-plane coordinates,  $(1/3)(C_{11} + 2C_{12} + 4C_{44})^*$  equals approximately one-half of  $(1/3)(C_{11} - C_{12} + C_{44})$ . Thus, the out-of-plane vibrations are significant, indicating significant climb or kink motion. Surprisingly, in (110)-glide-plane coordinates, both shear modes  $(1/2)(C_{11} - C_{12})^*$  and  $C_{44}$  are similar and approximately equal to  $(1/3)(C_{11} - C_{12} + C_{44})^*$ .

**P14-2**  
**AUSTENITIC-MARTENSITIC INTERFACES DAMPING MEASURE IN SHAPE MEMORY ALLOYS. REALIZATION OF A CYCLIC TENSILE MACHINE**

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The aim of this paper is to present a system to measure the austenitic - martensitic interfaces friction during the martensitic transformation of a CuZnAl stress induced monovariant.

A tensile machine, stress or strain controled, has been designed. The applied strain varies like a sine wave around a given mean value. The strain is measured by means of an extensometer fixed on the shape memory alloy sample. The load and strain signals are measured by the electronic system of a forced oscillation pendulum. Thus, from these data acquisitions, the energy dissipated during the martensitic transformation is calculated at each stress cycle. By using this set up the internal friction is measured from the area of tensile stress strain loops, with a cycling frequency of about 0,1 Hz.

The measures are carried out with a CuZnAl single crystal that is transformed under stress to a martensitic monovariant from an austenitic one.

During the transformation it is possible to count visually the austenitic - martensitic interfaces number and then to calculate the interface area.

From the damping measure and the interface area determination, the dissipated energy per austenitic - martensitic interface area unit is deduced. So doing the evolution of the damping can be monitored as the phase transformation proceeds.

**P14-3**  
**STRESS RELAXATION AND DAMPING BEHAVIOUR IN PRECIPITATION HARDENABLE ALLOYS**

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In various precision mechanical devices like high resolution electrodynamic balances engineering materials with low stress relaxation strength ( $\Delta$ ) are needed especially for spring applications. In order to obtain small spring constants one has to choose small dimensions requiring a high yield strength. Precipitation hardening is known to be effective for the increase of yield strength even at higher temperatures. Unfortunately only little is known about the effect of precipitates on stress relaxation and damping.

Therefore we measured the anelastic portion of the stress relaxation and the logarithmic decrement ( $\delta$ ) of flexural vibrations of copper-beryllium, nickel base and magnesium base precipitation hardenable alloys near room temperature. The samples have been subjected to various thermomechanical treatments in order to change the dislocation density and the degree of precipitation.

The time dependent stress has been determined using bending samples, which have been clamped on one side, with the aid of an electrodynamic balance for strains  $10^{-5}$ - $10^{-4}$ , times 3s-3000s and temperatures 20°C-40°C.  $\delta$  has been measured at room temperature using free decaying flexural vibrations of the same single clamped specimens excited in fundamental mode. The covered strain range was  $10^{-6}$ - $10^{-3}$ . The frequency ranged from 30 Hz to 300 Hz.

Specimens in supersaturated solid solution state deformed and undeformed have been heat treated isochronal (1h) step by step raising the temperature from room temperature up to below melting temperature. After each heat treatment the specimens have been quenched in cold water.  $\Delta$ ,  $\delta$  and hardness (HV) have been measured immediately after heat treatment.

The effect of precipitates on the properties of internal friction is not uniform. During precipitation  $\Delta$  changes especially for longer relaxation times. In all specimens  $\Delta$  recovers after thermal or mechanical treatment noticeable depending on the precipitation state and temperature over several days. The strength of recovery is, more than the relaxation strength, depending on the precipitation state. The logarithmic decrement is superimposed by a thermal current effect scaling with frequency and the square of sample thickness. The dependence of  $\delta$  on strain is similar to the one predicted by the Granato-Lücke-theory.

**P14-4**  
**NEAR-PLANE-WAVE ULTRASONIC ABSORPTION AND PHASE VELOCITY IN THIN SAMPLES OF ADHESIVE POLYMERS**

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This paper presents a novel wide bandwidth pulse transmission technique to measure ultrasonic absorption and phase velocity as functions of frequency (1 to 50 MHz) in thin (50  $\mu$ m) samples of adhesive set between float glass substrates. Thick PZT transducers are excited by very short pulses (120 V x 2 ns) and transmit correspondingly short disturbances into the adhesive. The signals received consist of time resolvable and successively dispersed acoustic reverberations from the bond layer. They are digitised at 1GHz and approximately corrected for the effects of transducer insertion and transient radiation coupling between the transducers. Frequency domain methods are applied to estimate absorption coefficient, phase velocity and the real and imaginary parts of the plane wave modulus as functions of frequency. Experimental results are given which show typical adhesive properties, and changes to these during cure and as functions of service temperature. These experiments indicate that the acoustic behaviour of a number of adhesives can be modelled by a relaxation process with a single time constant. A simple spring-dashpot model for an anelastic solid provides a mechanistic equivalent to the observed relaxation.

**P14-5**  
**NDE MECHANIC PROPERTY OF SOLID WITH LASER ULTRASONICS**

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Laser generating and receiving ultrasound or laser generating and capacitor transducer receiving ultrasound have potential for a wide range of non-contact ultrasonic measurements and have therefore received considerable interest over the past decade. The physical principles, and application to industry using nano-second (NS) laser ultrasonics have been reviewed by Hutchins and Scruby. The nondestructive testing (hereafter NDT) of microstructures, primarily layered structures such as combinations of thin films ranging in thickness from 100 Å to a few microns and distribution of electric charge in dielectric film using pico-second (PS) laser ultrasonics has been described by Maris et. al., and Sessel. In this paper the possibility of NDE of ultrasonic property of some special solid using laser ultrasonics technique is described. Firstly, a laser acoustic spectroscopy in our laboratory was introduced. Secondly, the dependence of the spectra of sources and the efficiencies on the properties and surface situation of materials were given. Thirdly, The examples of NDE worked in our laboratory for some special materials such as composites, polymer, small anisotropic crystal and nano-meter materials using laser ultrasonic technique were present. Finally what research must do in the future was discussed too.

**P14-6**  
**LOW COST VIBRATING REED MECHANICAL SPECTROMETER USING CAPACITIVE CURRENT DETECTION**

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A new, low cost, vibrating reed mechanical spectrometer has been developed. The set-up consists of a pulse train generation system for capacitive excitation of the specimen in one of its flexural vibration modes and of a capacitive current measuring and amplification circuit for detection of the specimen oscillation. The resonance frequency and the mechanical energy dissipation are derived by numerical analysis of the measured freely decaying vibration. Multiple electrode operation allows measurements of several flexural resonance modes with the same specimen. A special feature of the proposed set-up is the use of off-the-shelf equipment and easy-to-use software, resulting in fairly inexpensive construction, yet allowing good precision. Performance will be illustrated with one or two informative examples.

**P14-7**

**A COMPARISON BETWEEN COMPUTER MODELING AND ACOUSTIC SCANNING OF THE STRESS FIELD OF A DIAMETRALLY COMPRESSED DISC**

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It has been shown in our earlier work that by the lowering the lens in an acoustic microscope, mode-converted, polarized waves can be observed. The amplitude of these polarized modes depends on value of the acting stress, because the interface between a solid and water acts like a wave polarization analyzer. By using the scanning capability of the microscope, the stress intensity pattern can be displayed and compared with a computer-generated stress-distribution. Comparison between the calculated sum and difference of the principal stresses acting in the compressed disc and the acoustic images obtained by use of shear and longitudinal modes will also be discussed. A simple model explaining observed similarities is derived.

**P14-8**

**A FEASIBILITY STUDY FOR NON-CONTACT ACOUSTIC IMAGING OF STRESS DISTRIBUTION IN METALS**

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When polarized shear waves enter a stressed region of metal they experience birefringence effect that depends on the values and the directions of the acting principal stresses in that volume. By using an electromagnetic acoustic transducer (EMAT) to generate and detect polarized shear waves that propagate through stressed and unstressed regions of metal plates, we have demonstrated feasibility of displaying stress distributions in planar materials. The electromagnetic transducer acts as an analyzer for the receiving differently polarized shear acoustic waves from the stressed region. The monitored amplitude of that signal in acoustic imaging depends on the direction of polarization of shear modes which align along the directions of the principal stresses acting in the material. The outcome of this research can provide a non-contact scanning, stress-imaging technique, directly applicable to metals and indirectly to other materials.

P14-9

AMPLITUDE DOMAIN MECHANICAL SPECTROSCOPY. A POSSIBLE WAY TO A SYSTEMATIC APPROACH

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While mechanical spectroscopy analysis has been mostly carried out in the *frequency* (or temperature) domain, the *amplitude* domain has been, till now, far less considered.

The possible reasons for the above, will be summarized and the outstanding potentialities of the *Amplitude Domain Mechanical Spectroscopy (ADMS)*, as an investigation tool in *Materials Science and Engineering*, briefly outlined. The question will be examined with regard, not only to pure and "simple" laboratory specimens but having in mind the complex microstructure of the ordinary technical materials too.

Concerned mainly with elastic and dissipative phenomena, the ADMS should clearly be related to the shape of the curves expressing the amplitude dependence of elastic modulus and internal friction; namely, the peaks of the ADMS-spectra should be relevant to corresponding singularities or peculiarities in the so called Amplitude Dependence Curves (ADC's). A method for analysing such ADC's will thus be reported and examples of Damping Spectra vs Amplitude will be given (as obtained from experimental ADC's).

The generalized qualitative model representing the basis for the above processing method will be resumed and an attempt to justify it, in terms of microstructure elements and properties, will be made. An attempt will be made to indicate problems, in the field of the analytical treatment of "nonlinear phenomena", whose resolution seems to be a priority to attaining a fully quantitative model.

P14-10

INTERNAL FRICTION AND FREQUENCY MEASUREMENT SYSTEM BY VELOCITY METHOD

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The internal friction and frequency measurement system was made and coupled to a conventional torsion pendulum using an own made and low cost interface. The velocity method was implemented instead of the traditional and cumbersome amplitude decay method. Time intervals measurements (for velocity measurements) spent by a He-Ne laser spot to sweep a distance between two phototransistors mounted suitably in one screen were taken. The linear and logic ICs were coupled to trigger a one bit input of microcomputer which works as a CPU cycle counter programmed in Assembly. The counting is stored in RAM type memory and saved in the floppy disk. This method has advantages for later automation of the system for internal friction less than  $10^{-4}$ , with low rate temperature change. The frequency measurement is done by period measurements with addition of an extra logic circuit. The results of frequency as a function of temperature is important to identify Debye peaks in the former measurements. The details are described with aid circuit and a block diagram. The torsion pendulum can operate in temperatures from 77 to 700 K and frequencies between 0.1 and 10 Hz. (Financial support: CNPq and FUNDUNESP).

**P14-11**  
**DETERMINATION OF ULTRASONIC ATTENUATION IN SMALL SAMPLES OF SOLID MATERIAL BY SCANNING ACOUSTIC MICROSCOPY WITH PHASE CONTRAST**

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The attenuation of solid material or droplets of viscous liquids deposited on planar substrates is measured in the frequency regime of 300 MHz to 1.2 GHz by scanning acoustic microscopy with phase contrast. The observed spatial variations of the phases and amplitudes of the ultrasonic waves reflected from the objects contain sufficient information to determine the attenuation and velocity of ultrasonic waves in acoustically isotropic samples of minimal thickness of about 1  $\mu\text{m}$  and lateral extension down to 10  $\mu\text{m}$ . Additionally the spatial variation of the thickness of the objects can be determined with a resolution of about 1 nm. The basic principles are discussed and an approximative treatment is presented together with typical results.

**P14-12**  
**AN APPROACH TO HIGHER DAMPING CAPACITY: A COMPARISON OF MATERIAL DAMPING WITH COMPUTER-CONTROLLED DAMPING**

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An approach to higher damping by positively utilizing computer-aided control techniques has been investigated from comparisons of the relative magnitudes of the active(computer-controlled) damping and the material damping one.

The computer-controlled damping system is constructed with Brass plate (3x35x300mm<sup>3</sup>) as vibration-controlled objectives and a feedback controller to supply the dissipation energy to the Brass beam. The control system consists of a strain gauge type vibration sensor, an electro-magnetic type actuator concretely generating the vibration damping force, and a PC9801 type micro-computer. As the control law giving rise to the damping force, an adaptive model-following control scheme is employed, while the ferro-magnetic type high damping metal SIA is adopted for the reference.

The active damping effect increases the damping capacity of the beam vibration system from  $\delta_0=0.012$ (logarithmic decrement value in free vibration) to  $\delta_c=0.3248$ (total damping), and the value of  $\delta_c$  amounts to about 2.24 times the maximum damping  $Q^{-1}=0.023$  observed in SIA.

**P14-13**  
**MEASURING APPLICATIONS OF THE SCANNING ACOUSTIC MICROSCOPE (SAM)**

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Most of Internal Friction and Ultrasonic Velocity and Attenuation measurements are performed using bulk samples. Results are spatially averaged and obtained using one mode of vibration or propagation at a time.

Scannig Acoustic Microscopy allows one to make similar measurements locally. One of the applications is to measure separately elastic properties of the constituents of the composite materials. SAM may be also used to study the properties of thin layers. In layered media several modes of the Surface Acoustic Waves can be generated and their dispersion can be measured. Using the numerical inversion procedure, physical properties of the thin films are obtained [1].

Advantages and limitation of the technique will be discussed.

[1] Thin film characterisation using generalized Lamb wave and harmonic generation, P. Richard et al. - this conference.

**P14-14**  
**DIFFERENT ULTRASONIC ATTENUATION IN WELDS WITH NICKEL FILLING DETECTED BY SLAM**

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Electron beam welds with different contents of nickel like filling materials were observed by SLAM. The SLAM (Scanning Laser Acoustic Microscope) system uses the propagation of continuous plane ultrasonic waves at the frequencies of 10, 30 or 100 MHz to detect change in the structure, defects or different phases inside the sample. A scanning laser beam is used as an ultrasound detector by means of sensing the displacements (rippling) of the surface created by the ultrasonic waves. In this way it is possible to display in real time an acoustic pattern that corresponds to the sample surface displacement, carrying information about the material properties along the whole sample thickness.

Acoustic images of welds with 0, 100  $\mu\text{m}$  and 180  $\mu\text{m}$  of Nickel filling in Fe460 metal base were analysed. A different scattering by grains would be the cause of the different ultrasonic attenuation along the welds. In fact SAM (Scanning Acoustic Microscopy), using Rayleigh waves (1300 MHz), and optical images have shown a different surface structure and morphology in the welds. Calculations of Rayleigh scattering and stochastic scattering confirm this hypothesis.

Therefore microindentation measurements and ultrasonic attenuation result to follow the same profile along different weld lines.

In conclusion it is possible to investigate structural changes and mechanical properties nondestructively by SLAM.

**P14-15**  
**MEASUREMENT OF STRESS RELAXATION STRENGTH IN ENGINEERING MATERIALS FOR  
HIGH PRECISION MECHANICAL APPLICATIONS**

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Mechanical relaxation of engineering materials in high resolution mechanical devices can be a serious problem. In electrodynamic balances eg. it leads to a time dependent drift of the electric current through a coil compensating the weight by a magnetic force. Without time dependent correction of this signal the reading of the balance, which is proportional to this current, is also changing with time. This behaviour can be avoided by the use of materials with very low relaxation strength for certain elements in the balance.

The problem of measuring relative relaxation strength in the order of  $10^{-4}$  -  $10^{-2}$  has been solved itself by using an electrodynamic balance with a resolution of  $10^{-6}$  (1 mg res., 2 kg max.). It has been modified in order to load the free side of a bending sample clamped on one side by lifting or sinking the scale of the balance which is connected to the free side of the sample by a weight fully supported by the scale and the sample. After loading the sample its relaxing stress can be calculated using the time dependent reading of the balance. This measuring technique is well suited to the problem because the measuring conditions like stress level, stress resolution, range of measuring time, and mode of relaxation are the same as the relaxation conditions in the balance. The balance reading is used to calculate the anelastic part of the stress relaxation of the sample in the measured range of time beginning at  $t = 3$  s.

**P14-16**  
**PIEZOLETRIC METHOD OF DETERMINING FLEXURAL DAMPING AT FREQUENCIES OF 0.5  
TO 10 kHz**

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The use of a quartz resonant piezoelectric oscillator is an established method of generating longitudinal and torsional strain waves at frequencies in the region of 30 to 180 kHz. This technique is used for measuring internal friction, piezoelectric defects and strain effects in solids.

This paper presents a composite oscillator system vibrating in the flexural mode with the piezoelectric element producing strain waves in the range of 0.5 to 10 kHz. The oscillating strain varies from extension to compression across the crystal thickness.

By using different plating on the faces, two conventional 40 kHz longitudinal piezoelectric crystals can be operated at 2.7 kHz in the flexural mode as part of a two-component drive/gauge unit. The system can then be extended to contain a sample element. However, the resonant wave-form is not a simple sinusoid, thus the extension of a two-component composite oscillator to three or more components requires some care and experimental verification.

The development of the electrical-mechanical equivalent circuit for the flexural system was derived from the flexural vibration theory originally developed by Raleigh. The resulting circuit for the flexural system was tested experimentally for two-, three-, and four-component oscillators.

The results indicate that the method is of general applicability and offers a useful tool in the armoury of the researcher using acoustic techniques to study material properties and/or produce an oscillating strain or displacement in a controlled manner at frequencies of 0.5 to 10 kHz.

**P14-17**

**NONLINEAR SPATIAL FREE DAMPING VIBRATIONS OF SUSPENSION COMBINED SYSTEMS**

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Free vibrations of a suspension combined system may produce intensive energy exchange between two partial systems, for example, between vertical and torsional modes, with the result that the maximum (minimum) amplitude of one mode is accompanied by the minimum (maximum) amplitude of the other mode. Such an energy transform is observed at a one-to-one or two-to-one internal resonance, when the natural frequency of the vertical vibrations approximately equal or twice as large as that of the torsional vibrations. When investigating this phenomenon, a low damping capacity of suspension combined systems should be taken into account. This circumstance cannot be ignored in the design of suspension and cable-stayed bridges.

In this paper, the influence of a small viscosity upon the behavior of vibration motions in a suspension combined system is analyzed for one-to-one and two-to-one internal resonance, in so doing the energy dissipation of such a system is defined by an exponential dependence. A set of governing equations describing nonlinear free damping vibrations is obtained. The method of multiple scales is used as a method of solution. Results of the numerical investigations on free damping vibrations of Golden Gate Bridge under internal resonance conditions are presented.

**P14-18**

**LOCALIZED POLYMERS CURING WITH CO<sub>2</sub> LASER AND APPLICATION: DEMAND - CURING AND 3D - LITHOGRAPHY**

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We report on new technique for localized polymer curing with 0,1 mm resolution in all 3D-axis, using CO<sub>2</sub> laser. The technique was developed from a theoretical model that explains the complex phenomena of thermal pulse propagation after laser irradiation, and how to control the expansion of laser deposited heat. The paper discusses both theoretical and experimental results, demand-curing for localized glueing and 3D-lithography, for rapid prototyping of parts for the industry.

P14-19

A SUGGESTION ON THE FIFTH GENERATION OF KE'S PENDULUM

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The process of development of the torsion pendulum, from the Coulomb's torsion scale to the modern multi-function Ke's pendulum made in China, was analysed. It seems to us that there are still a series of shortcomings, for instance, low speed of measurement, narrow frequency range, high background damping, inhomogeneous strain etc., and only equipment parameters of internal friction can be obtained directly. There is no standard of the measurement of internal friction and it is difficult to build a precise data base of material parameters for internal friction even use the best torsion pendulum. In addition, the anelasticity associated with the dislocation and boundary were found to be of the non-steady, non-Debye and non-linear peculiarities. Therefore, both the internal friction peak and the background are of equal importance. An absolute measurement of the material parameters of the internal friction is appreciated.

A computer simulation showed that by using state-of-the-art of IC, and the high speed CPU, the accuracy, upper limit of frequencies and speed of measurement of non-linear or non-steady internal friction parameter of materials could be improved substantially either for force vibration or for the free decay methods. For the fifth generation of Ke's equipment, entirely single freedom oscillation, homogeneous strain, to be able to anneal or bias in situ, wide frequency range, high speed and high accuracy of measurement would be essential. A few possible schemes are proposed, in which either non-resonant quartz under static press stress or a longitudinal pendulum fixed at two ends would be used with the high speed data acquisition card such as ADA3100 and an accelerator card, INMOS transputer T900 or better, INTEL A860, on IBM PC microcomputer system.

The best place for the low frequency sound adsorption spectrum laboratory would be on the reduced gravity environment of space. This would be a new task for the remote science. An international cooperation is worthwhile.

P14-20

SCANNING ACOUSTIC IMAGING OF RESIDUAL STRESSES IN CERAMICS

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This paper presents the application of a scanning acoustic microscopy technique for the detection and evaluation of the internal residual stresses around the indentation from microhardness test. It is based upon the sensitivity of polarized acoustic modes to the local elastic anisotropy caused by stress. The analogy between photoelastic and acoustic techniques are discussed, and the quantitative stress analysis is presented. The studies of transmitted, reflected and received pulses monitored in low frequency experiments explains the stress pattern seen at high frequencies.

This is a practical application of a previously proposed method of imaging stresses by scanning acoustic microscopy. The two cases discussed are: the low frequency (10 MHz) acoustic imaging of the distribution of the internal stresses around an indentation in a silicon carbide sample (9mm thick), and more complex high frequency (200-800 MHz) stress visualization of a Vicker's indentation test in a silicon nitride sample (2mm thick). In both cases, the maximum shear elastic stress surrounding the indentations are imaged using polarized acoustic modes and the values of the stresses are calculated based on the image evaluation of the shear stress cone diameter.

**P14-21**  
**ANELASTIC PROPERTIES OF RESONANT TRANSDUCERS FOR CRYOGENIC GRAVITATIONAL WAVE ANTENNAS**

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Low anelastic losses are essential in order to obtain a good sensitivity of resonant cryogenic gravitational wave antennas. In the most common transduction scheme, the energy left in the antenna by the gravitational wave is transferred to a small mass having the same resonant frequency. In this way, it is possible to convert the mechanical excitation in an electrical signal with higher efficiency. The small resonator coupled to the large detection mass should have anelastic characteristics as good as those of the antenna, in order not to degrade the sensitivity of the apparatus. Results of anelastic relaxation measurements performed on resonators of Al 5056, Ti grade 2 and Steel NiCrMo4 at a frequency of 1 kHz from room temperature down to 1.5 K will be presented.

**P14-22**  
**ANELASTIC AND ELASTIC PROPERTIES OF A SYNTHETIC MONOCRYSTAL OF BISMUTH GERMANATE ( $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ )**

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We have investigated the elastic and anelastic properties of a synthetic Bismuth Germanate monocrystal with the perspective to use it as a building material for antennas and transducers for gravitational wave detection. The elastic and anelastic response of a small rod (20 cm length, 2 cm diameter) excited by piezoelectric ceramics glued on its surface has been observed from 300 K to 10 K, at frequencies of 10, 30 and 50 kHz, corresponding to the first, third and fifth longitudinal harmonic respectively. We have obtained the behaviour of the Young Modulus, of the Poisson Ratio and of the anelastic relaxations as a function of temperature.

**P14-23**  
**A NEW APPARATUS FOR LOW FREQUENCY MECHANICAL SPECTROSCOPIC MEASUREMENTS**

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A new apparatus has been developed for complex shear modulus measurements in the 0.001 -1000 Hz frequency range.

Specimens in the form of flat bars are electrostatically driven by two electrodes which also serve to obtain the displacement by a capacitive method.

Due to the special form of the specimens, a pure torsional mode is excited and a low extra damping level observed.

That new apparatus thus appears as an over-simplified torsion pendulum with improved performances, well suited for tests on small samples of brittle materials. Insulating materials can also be tested after metal deposition on a non strained part of the specimen.

That new internal friction measurement method has been successfully used in "flux pinning" experiments in high Tc YBaCuO specimens.

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